Synthesis of Water-Tolerant Indium Homoenolate in Aqueous Media and Its Application in the Synthesis of 1,4-Dicarbonyl Compounds via Palladium-Catalyzed Coupling with Acid Chloride

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Supporting Information

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General methods

All acid chlorides were used directly without purification. Following commercial grade solvents and reagents were also used without further purification: indium (powder, -100 mesh, 99.99%, Aldrich chemicals), indium trichloride (strem chemical), DMA *(*Alfa Aesar chemical*), trans*-dichlorobis(triphenylphosphine)palladium (Alfa Aesar chemical).

Analytical thin layer chromatography (TLC) was performed using Merck 60 F254 precoated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm) on Spectroline Model ENF-24061/F 254 nm. Further visualization was possible by staining with acidic solution of ceric molybdate.

Flash chromatography was performed using Merck silica gel 60 with freshly distilled solvents. Columns were typically packed as slurry and equilibrated with the appropriate solvent system prior to use.

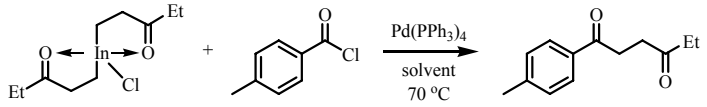
Infrared spectra were recorded on a Bio-Rad FTS 165 FTIR spectrometer. The oil samples were examined under neat conditions.

High Resolution Mass (HRMS) spectra were obtained using Finnigan MAT95XP GC/HRMS (Thermo Electron Corporation).

Proton nuclear magnetic resonance spectra (^{1}H NMR) were recorded on a Bruker Avance DPX 300 and Bruker AMX 400 spectrophotometer ($CDCl_3$ as solvent). Chemical shifts for ^{1}H NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-*d* (δ 7.2600, singlet). Multiplicities were given as: s (singlet); d (doublet); t (triplet); q (quartet); or m (multiplets). The number of protons (n) for a given resonance is indicated by nH. Coupling constants are reported as a *J* value in Hz. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-*d* (δ 77.03, triplet).

Optimization of reaction conditions

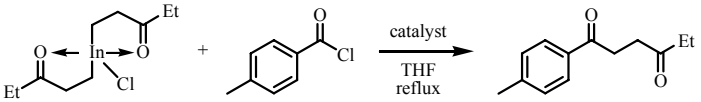
Table 1. Optimization of Reaction Conditions[a]



entry	solvent	yield (%)[b]
1	**THF** (reflux)	**81**
2	dioxane	79
3	dioxane (100 °C)	74
4	*t*-BuOMe (reflux)	54
5	DMA	67
6	benzene	<20%
7	CH_3CN	trace
8	CH_3NO_2	trace
9	$ClCH_2CH_2Cl$	4
10	DMF	8
11	DMSO	trace

[a] Unless otherwise noted, the reactions were carried out at 70 °C for 24 h using indium homoenolate (0.3 mmol), acid chloride (0.5 mmol), and $Pd(PPh_3)_4$ (0.025 mmol) in various organic solvents (3 mL). [b] Isolated yield.

Table 2. Optimization of Reaction Conditions[a]



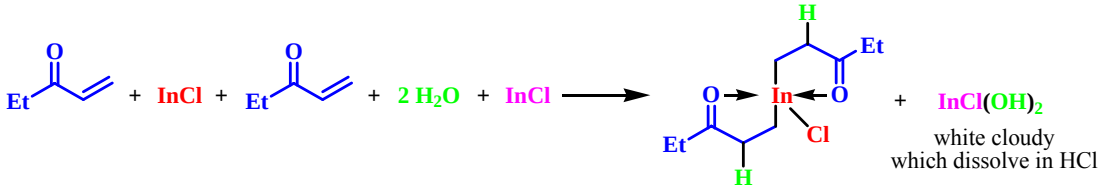
entry	catalyst	yield (%)[b]
1	$PdCl_2$ (5 mol%), PPh_3 (10 mol%)	84
2	$PdCl_2$ (5 mol%), DPEPhos (10 mol%)	56
3	$Pd(OAc)_2$ (5 mol%), PPh_3 (10 mol%)	84
4	**$PdCl_2(PPh_3)_2$ (5 mol%)**	**87**
5	$Pd(dppf)Cl_2$ (5 mol%)	68
6	$Pd(PhCN)_2Cl_2$ (5 mol%), PPh_3 (10 mol%)	65
7	$Pd(MeCN)_2Cl_2$ (5 mol%), PPh_3 (10 mol%)	84
8	$Pd_2(dba)_3$ (2.5 mol%), PPh_3 (10 mol%)	84
9	$Pd(PPh_3)_4$ (5 mol%)	81

[a] Unless otherwise noted, the reactions were carried out at reflux for 24 h using indium homoenolate (0.3 mmol), acid chloride (0.5 mmol), and various palladium catalysts (0.025 mmol) in THF (3 mL). [b] Isolated yield.

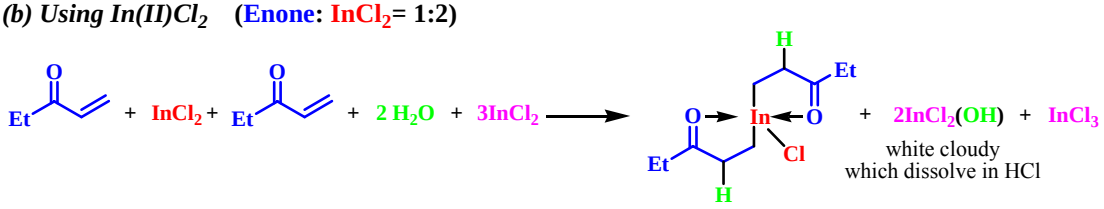
Equations account for the theoretical amount of InCl and $InCl_2$ which should be used in the formation of indium homoenolate

It should be noted that, after reaction a white cloudy solution was observed and it might dissolve in aq. HCl, which indicates the formation of an indium hydroxide complex with a formula of $InCl_x(OH)_y$ (x + y =3). According to the following equation, one equiv. of InCl is enough for the complete conversion of enone to homoenolate; in the meantime, two equiv. $InCl_2$ is necessary for the conversion of one equiv. enone to the desired product. The use of slightly excess of InCl (1.2 eqiv.) and $InCl_2$ (2.4 equiv.) in the reaction also confirmed our hypothesis that complete conversion of enone was observed as indicated by crude NMR analysis.

(a) Using In(I)Cl **(Enone: InCl = 1:1)**



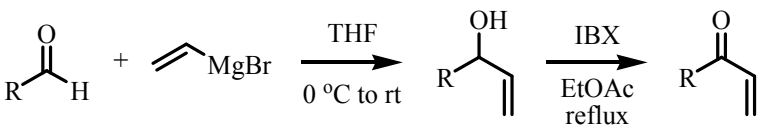
(b) Using In(II)Cl$_2$ **(Enone: $InCl_2$= 1:2)**



Experimental procedure

General procedure for the synthesis of enone

Method 1: Grignard reaction followed by IBX oxidation

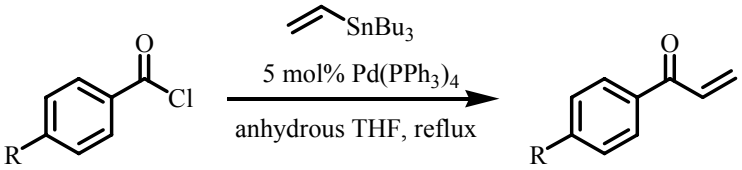


Grignard reaction:[1] A solution of aldehyde (20 mmol, 1 equiv.) in anhydrous THF (40

mL) was stirred for 10 minutes under nitrogen atmosphere at 0 °C. Vinylmagnesium bromide (40 mmol, 2 equiv., 1 M in THF) was added dropwise and the reaction was stirred overnight. After reaction, it was quenched with saturated aqueous NH_4Cl solution and extracted with diethyl ether. The combined organic layers were washed with brine, dried over anhydrous $MgSO_4$, filtered, and concentrated in *vacuo*. The residue was subjected to silica gel column chromatography using hexane and EtOAc as eluant to afford the desired allylic alcohol.

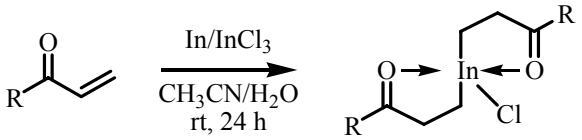
IBX oxidation:[2] IBX (12 mmol, 1.2 equiv) was added portionwise into a solution of the above allylic alcohol (10 mmol, 1 equiv) in ethyl acetate and stirred overnight at reflux. The mixture was then filtered on celite and further washed with ethyl acetate. The combined filtrate was washed with brine, dried over anhydrous $MgSO_4$, filtered, and concentrated in *vacuo*. The residue was subjected to silica gel column chromatography using hexane and EtOAc as eluant to afford the desired enone.

Method 2: Palladium-catalyzed coupling of tributyl(vinyl)stannane with acid chloride[3]



To a solution of tributyl(vinyl)stannane (10 mmol, 1 equiv.) in anhydrous THF (50 mL) was added acid chloride (10 mmol, 1 equiv.) and tetrakis(triphenylphosphine) palladium (0.5 mmol, 0.05 equiv.) and stirred at reflux for two hours. The mixture was then diluted with diethyl ether (100 mL) and washed with aqueous potassium fluoride (30 mL x 3). The organic layer was then further washed with brine, dried over anhydrous $MgSO_4$ and concentrated in *vacuo*. The residue was subjected to silica gel column chromatography using hexane and EtOAc as eluant to afford the desired enone.

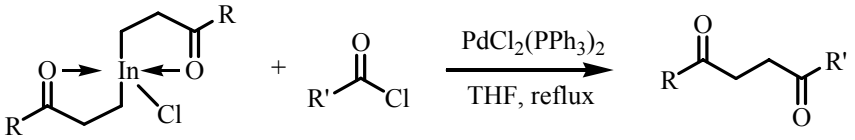
General procedure for the synthesis of indium homoenolate from enone



To a solution of enone (2 mmol, 1 equiv.) in CH_3CN:H_2O (1:1, 10 mL) was added indium (1.6 mmol, 0.8 equiv.) and $InCl_3$ (0.8 mmol, 0.4 equiv.). The reaction mixture was stirred vigorously at room temperature for 24 hrs. After reaction, the mixture was extracted

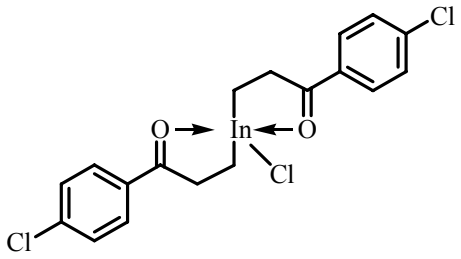
with dichloromethane (20 mL x 5), washed with brine and dried over $MgSO_4$. It was then concentrated in *vacuo* and the residue was purified by silica gel column chromatography using MeOH/CH_2Cl_2 as eluant to afford the desired indium homoenolate.

General procedure for the palladium-catalyzed coupling reaction of indium homoenolate with acid chloride



To a solution of indium homoenolate (0.3 mmol) in anhydrous THF (3 mL) was added acid chloride (0.5 mmol, 1.0 equiv) and $PdCl_2(PPh_3)_2$ (0.025 mmol, 0.05 equiv). The reaction mixture was stirred vigorously at reflux for 24 h. After reaction, solvent was removed in *vacuo* and the residue was directly purified by silica gel column chromatography using EtOAc/hexane as eluant to afford the desired product of 1,4-dicarbonyl compound.

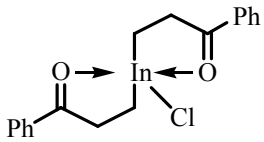
Spectroscopic data of product



Indium homoenolate 1a

Prepared according to the general procedure with enone (0.333 g, 2 mmol), indium (0.184 g, 1.6 mmol), and indium(III) chloride (0.177 g, 0.8 mmol) in CH_3CN (5 mL) and H_2O (5 mL) to afford the title compound in 75% isolated yield (0.365 g, 0.75 mmol).

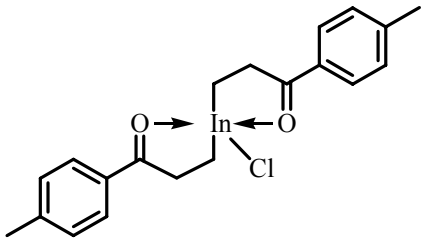
^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.09 (t, J = 6.90 Hz, 4H), 3.59 (t, J = 6.90 Hz, 4H), 7.37 (d, J = 8.50 Hz, 4H), 7.91 (d, J = 8.50 Hz, 4H) ppm. ^{13}C NMR ($CDCl_3$, 125 MHz): δ 10.0, 35.8, 128.8, 129.9, 134.0, 140.3, 206.3 ppm. HRMS (ESI, m/z): $[M\text{-}Cl]^+$, calcd. for $C_{18}H_{16}O_2Cl_2In$: 448.9566, found: 448.9556. FTIR (KBr, neat): ν 1651 cm^{-1}.



Indium homoenolate 1b

Prepared according to the general procedure with enone (0.264 g, 2 mmol), indium (0.184 g, 1.6 mmol), and indium(III) chloride (0.177 g, 0.8 mmol) in CH_3CN (5 mL) and H_2O (5 mL) to afford the title compound in 53% isolated yield (0.222 g, 0.53 mmol).

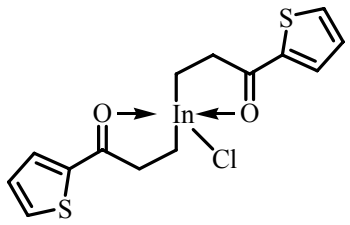
^{1}H NMR (500 MHz, $CDCl_3$): δ 1.04 (t, J = 6.95 Hz, 4H), 3.67 (t, J = 6.95 Hz, 4H), 7.48 (t, J = 7.56 Hz, 4H), 7.62 (t, J = 7.41 Hz, 2H), 8.08 (d, J = 8.10 Hz, 4H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 7.3, 35.9, 128.7, 128.9, 134.3, 135.7, 208.6 ppm. HRMS (ESI, m/z): $[M\text{-}Cl]^+$, calcd. for $C_{18}H_{18}O_2In$: 381.0346, found: 381.0353. FTIR (KBr, neat): ν 1636 cm^{-1}.



Indium homoenolate 1c

Prepared according to the general procedure with enone (0.292 g, 2 mmol), indium (0.184 g, 1.6 mmol), and indium(III) chloride (0.177 g, 0.8 mmol) in CH_3CN (5 mL) and H_2O (5 mL) to afford the title compound in 89% isolated yield (0.395 g, 0.89 mmol).

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.04 (t, J = 7.01 Hz, 4H), 2.38 (s, 6H), 3.60 (t, J = 7.01 Hz, 4H), 7.22 (d, J = 8.09 Hz, 4H), 7.92 (d, J = 8.24 Hz, 4H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 8.2, 21.6, 35.6, 128.8, 129.2, 133.2, 145.1, 207.7 ppm. HRMS (ESI, m/z): $[M\text{-}Cl]^+$, calcd. for $C_{20}H_{22}O_2In$: 409.0659, found: 409.0658. FTIR (KBr, neat): ν 1636 cm^{-1}.

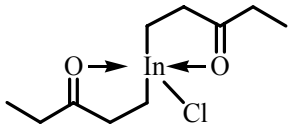


Indium homoenolate 1d

Prepared according to the general procedure with enone (0.276 g, 2 mmol), indium (0.184 g, 1.6 mmol), and indium(III) chloride (0.177 g, 0.8 mmol) in CH_3CN (5 mL) and H_2O (5 mL) to afford the title compound in 81% isolated yield (0.348 g, 0.81 mmol).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.03 (t, J = 7.60 Hz, 4H), 3.58 (t, J = 7.60 Hz, 4H), 7.16 (dd, J = 4.84, 3.95 Hz, 2H), 7.73 (dd, J = 4.90, 0.95 Hz, 2H), 7.91 (dd, J = 3.90, 1.05 Hz, 2H) ppm. ^{13}C NMR ($CDCl_3$, 125 MHz): δ 7.9, 36.5, 128.5, 133.9, 135.7, 142.1, 200.7 ppm. HRMS

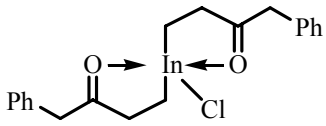
(ESI, m/z): $[M\text{-}Cl]^+$, calcd. for $C_{14}H_{14}O_2S_2In$: 392.9474, found: 392.9469. FTIR (KBr, neat): *v* 1624 cm^{-1}.



Indium homoenolate 1e

Prepared according to the general procedure with enone (0.168 g, 2 mmol), indium (0.184 g, 1.6 mmol), and indium(III) chloride (0.177 g, 0.8 mmol) in CH_3CN (5 mL) and H_2O (5 mL) to afford the title compound in 82% isolated yield (0.263 g, 0.82 mmol).

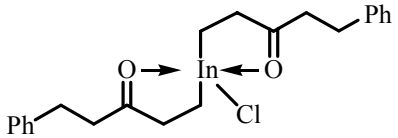
^1H NMR (500 MHz, $CDCl_3$): δ 0.83 (t, *J* = 7.16 Hz, 4H), 1.09 (t, *J* = 7.36 Hz, 6H), 2.52 (q, *J* = 7.36 Hz, 4H), 3.04 (t, *J* = 7.16 Hz, 4H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 7.9, 8.4, 35.6, 39.7, 221.5 ppm. HRMS (ESI, m/z): $[M\text{-}Cl]^+$, calcd. for $C_{10}H_{18}O_2In$: 285.0346, found: 285.0346. FTIR (KBr, neat): *v* 1674 cm^{-1}.



Indium homoenolate 1f

Prepared according to the general procedure with enone (0.292 g, 2 mmol), indium (0.184 g, 1.6 mmol), and indium(III) chloride (0.177 g, 0.8 mmol) in CH_3CN (5 mL) and H_2O (5 mL) to afford the title compound in 59% isolated yield (0.262 g, 0.59 mmol).

^1H NMR ($CDCl_3$, 500 MHz): δ 0.77 (t, *J* = 7.04 Hz, 4H), 3.08 (t, *J* = 7.04 Hz, 4H), 3.78 (s, 4H), 7.19 (d, *J* = 7.66 Hz, 4H), 7.26-7.34 (m, 6H) ppm. ^{13}C NMR ($CDCl_3$, 125 MHz): δ 9.2, 39.9, 49.2, 127.2, 128.7, 129.4, 133.4, 218.2 ppm. HRMS (ESI, m/z): $[M\text{-}Cl]^+$, calcd. for $C_{20}H_{22}O_2In$: 409.0659, found: 409.0654. FTIR (KBr, neat): *v* 1676 cm^{-1}.

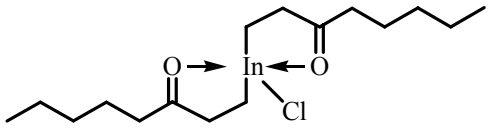


Indium homoenolate 1g

Prepared according to the general procedure with enone (0.320 g, 2 mmol), indium (0.184 g, 1.6 mmol), and indium(III) chloride (0.177 g, 0.8 mmol) in CH_3CN (5 mL) and H_2O (5 mL) to afford the title compound in 90% isolated yield (0.427 g, 0.90 mmol).

^1H NMR (500 MHz, $CDCl_3$): δ 0.83 (t, *J* = 7.11 Hz, 4H), 2.80 (t, *J* = 7.80 Hz, 4H), 2.92 (t, *J* = 7.47 Hz, 4H), 3.02 (t, *J* = 7.11 Hz, 4H), 7.14–7.20 (m, 6H), 7.26–7.29 (m, 4H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 8.3, 29.9, 40.6, 44.0, 126.3, 128.2, 128.6, 140.4, 220.2 ppm. HRMS (ESI,

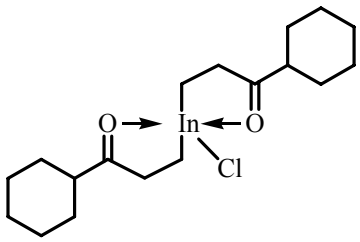
m/z): $[M\text{-}Cl]^+$, calcd. for $C_{22}H_{26}O_2In$: 437.0972, found: 437.0968. FTIR (KBr, neat): *v* 1682 cm^{-1}.



Indium homoenolate 1h

Prepared according to the general procedure with enone (0.252 g, 2 mmol), indium (0.184 g, 1.6 mmol), and indium(III) chloride (0.177 g, 0.8 mmol) in CH_3CN (5 mL) and H_2O (5 mL) to afford the title compound in 83% isolated yield (0.335 g, 0.83 mmol).

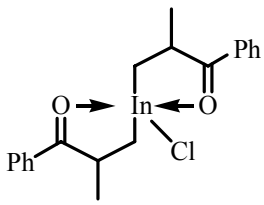
^1H NMR ($CDCl_3$, 500 MHz): δ 0.81 (t, *J* = 7.04 Hz, 4H), 0.87 (t, *J* = 7.02 Hz, 6H), 1.23-1.32 (m, 8H), 1.57-1.63 (m, 4H), 2.48 (t, *J* = 7.40 Hz, 4H), 3.04 (t, *J* = 7.04 Hz, 4H) ppm. ^{13}C NMR ($CDCl_3$, 125 MHz): δ 8.4, 13.8, 22.3, 23.7, 31.2, 40.2, 42.4, 221.1 ppm. HRMS (ESI, m/z): $[M\text{-}Cl]^+$, calcd. for $C_{16}H_{30}O_2In$: 369.1285, found: 369.1277. FTIR (KBr, neat): *v* 1678 cm^{-1}.



Indium homoenolate 1i

Prepared according to the general procedure with enone (0.276 g, 2 mmol), indium (0.184 g, 1.6 mmol), and indium(III) chloride (0.177 g, 0.8 mmol) in CH_3CN (5 mL) and H_2O (5 mL) to afford the title compound in 82% isolated yield (0.353 g, 0.82 mmol).

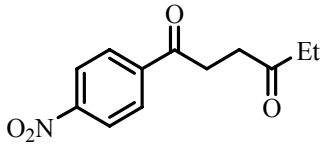
^1H NMR (400 MHz, $CDCl_3$): δ 0.78 (t, *J* = 7.06 Hz, 4H), 1.17–1.42 (m, 10H), 1.65–1.85 (m, 10H), 2.44 (tt, *J* = 11.10, 3.21 Hz, 2H), 3.07 (t, *J* = 7.06 Hz, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 7.3, 25.5, 25.7, 28.6, 38.5, 50.6, 224.4 ppm. HRMS (ESI, m/z): $[M\text{-}Cl]^+$, calcd. for $C_{18}H_{30}O_2In$: 393.1285, found: 393.1280. FTIR (KBr, neat): *v* 1676 cm^{-1}.



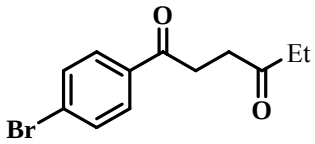
Indium homoenolate 1j

Prepared according to the general procedure with enone (0.292 g, 2 mmol), indium (0.184 g, 1.6 mmol), and indium(III) chloride (0.177 g, 0.8 mmol) in CH_3CN (5 mL) and H_2O (5 mL)

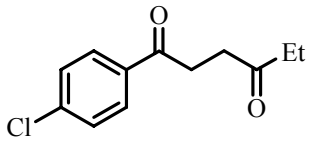
to afford the title compound in 74% isolated yield (0.329 g, 0.74 mmol).
^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.09-1.14 (m, 4H), 1.39 (t, *J* = 7.26 Hz, 6H), 4.30-4.35 (m, 2H), 7.48 (t, *J* = 7.74 Hz, 4H), 7.62 (t, *J* = 7.36 Hz, 2H), 8.07 (d, *J* = 8.18 Hz, 4H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 19.6, 19.8, 22.8, 39.8, 39.9, 128.8, 129.3, 134.3, 134.5, 134.5, 212.1, 212.1 ppm. HRMS (ESI, m/z): $[M\text{-}Cl]^+$, calcd. for $C_{20}H_{22}O_2In$: 409.0659, found: 409.0660. FTIR (KBr, neat): *v* 1636 cm^{-1}.



1-(4-Nitrophenyl)hexane-1,4-dione (2a)
Prepared according to the general procedure with indium homoenolate **1e** (0.096 g, 0.3 mmol), acid chloride (0.093 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 84% isolated yield (0.098 g, 0.42 mmol).
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.10 (t, *J* = 7.35 Hz, 3H), 2.58 (q, *J* = 7.36 Hz, 2H), 2.92 (t, *J* = 6.37 Hz, 2H), 3.31 (t, *J* = 5.82 Hz, 2H), 8.14 (d, *J* = 8.89 Hz, 2H), 8.30 (d, *J* = 8.88 Hz, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 7.7, 32.8, 35.6, 35.8, 123.7, 129.0, 141.1, 150.2, 197.3, 209.5 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{12}H_{14}NO_4$: 236.0923, found: 236.0924. FTIR (KBr, neat): *v* 1703, 1686 cm^{-1}.



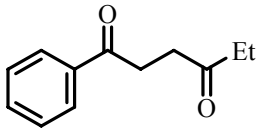
1-(4-Bromophenyl)hexane-1,4-dione (2b)
Prepared according to the general procedure with indium homoenolate **1e** (0.096 g, 0.3 mmol), acid chloride (0.110 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 61% isolated yield (0.082 g, 0.31 mmol; with ~5% impurity which cannot be purified by silica gel column chromatography).
^{1}H NMR (500 MHz, $CDCl_3$): δ 1.10 (t, *J* = 7.35 Hz, 3H), 2.56 (q, *J* = 7.35 Hz, 2H), 2.86 (t, *J* = 6.35 Hz, 2H), 3.24 (t, *J* = 6.10 Hz, 2H), 7.60 (d, *J* = 8.55 Hz, 2H), 7.85 (d, *J* = 8.56 Hz, 2H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 7.8, 32.3, 35.7, 36.1, 128.3, 129.6, 131.9, 135.4, 197.7, 210.0 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{12}H_{14}O_2Br$: 269.0177, found: 269.0187. FTIR (KBr, neat): *v* 1713, 1686 cm^{-1}.



1-(4-Chlorophenyl)hexane-1,4-dione (2c)[4]

Prepared according to the general procedure with indium homoenolate **1e** (0.096 g, 0.3 mmol), acid chloride (0.088 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 84% isolated yield (0.094 g, 0.42 mmol).

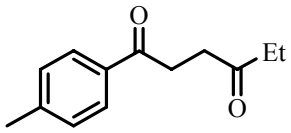
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.09 (t, *J* = 7.33 Hz, 3H), 2.55 (q, *J* = 7.36 Hz, 2H), 2.85 (t, *J* = 6.17 Hz, 2H), 3.23 (t, *J* = 5.83 Hz, 2H), 7.42 (d, *J* = 8.56 Hz, 2H), 7.91 (d, *J* = 8.58 Hz, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 7.7, 32.2, 35.6, 35.9, 128.7, 129.3, 134.9, 139.4, 197.3, 209.8 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{12}H_{14}O_2Cl$: 225.0682, found: 225.0686. FTIR (KBr, neat): *v* 1711, 1686 cm^{-1}.



1-Phenylhexane-1,4-dione (2d)[5]

Prepared according to the general procedure with indium homoenolate **1e** (0.096 g, 0.3 mmol), acid chloride (0.070 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 83% isolated yield (0.079 g, 0.42 mmol).

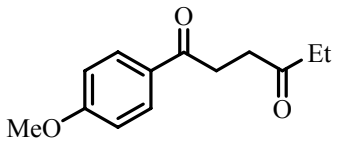
^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.10 (t, *J* = 7.36 Hz, 3H), 2.56 (q, *J* = 7.30 Hz, 2H), 2.86 (t, *J* = 6.44 Hz, 2H), 3.29 (t, *J* = 6.10 Hz, 2H), 7.44-7.48 (m, 2H), 7.54-7.58 (m, 1H), 7.97-8.00 (m, 2H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 7.8, 32.4, 35.7, 36.0, 128.0, 128.5, 133.1, 136.6, 198.7, 210.1 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{12}H_{15}O_2$: 191.1072, found: 191.1081. FTIR (KBr, neat): *v* 1715, 1686 cm^{-1}.



1-*p*-Tolylhexane-1,4-dione (2e)[6]

Prepared according to the general procedure with indium homoenolate **1e** (0.096 g, 0.3 mmol), acid chloride (0.077 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 87% isolated yield (0.089 g, 0.44 mmol).

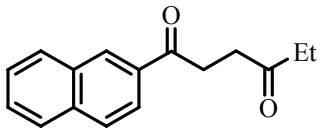
^{1}H NMR (300 MHz, $CDCl_3$): δ 1.09 (t, *J* = 7.35 Hz, 3H), 2.40 (s, 3H), 2.56 (q, *J* = 7.37 Hz, 2H), 2.84 (t, *J* = 6.45 Hz, 2H), 3.26 (t, *J* = 6.16 Hz, 2H), 7.25 (d, *J* = 8.07 Hz, 2H), 7.88 (d, *J* = 8.20 Hz, 2H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 7.8, 21.6, 32.3, 35.8, 36.0, 128.1, 129.2, 134.2, 143.8, 198.3, 210.1 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{13}H_{17}O_2$: 205.1229, found: 205.1228. FTIR (KBr, neat): *v* 1707, 1678 cm^{-1}.



1-(4-Methoxyphenyl)hexane-1,4-dione (2f)

Prepared according to the general procedure with indium homoenolate **1e** (0.096 g, 0.3 mmol), acid chloride (0.085 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 74% isolated yield (0.081 g, 0.37 mmol).

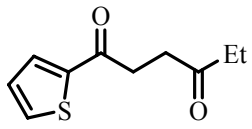
^{1}H NMR (500 MHz, $CDCl_3$): δ 1.09 (t, *J* = 7.35 Hz, 3H), 2.56 (q, *J* = 7.36 Hz, 2H), 2.83 (t, *J* = 6.35 Hz, 2H), 3.24 (t, *J* = 6.20 Hz, 2H), 3.86 (s, 3H), 6.93 (d, *J* = 8.75 Hz, 2H), 7.96 (d, *J* = 8.81 Hz, 2H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 7.8, 32.0, 35.8, 36.0, 55.4, 113.6, 129.7, 130.2, 163.4, 197.2, 210.3 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{13}H_{17}O_3$: 221.1178, found: 221.1180. FTIR (KBr, neat): *v* 1705, 1668 cm^{-1}.



1-(Naphthalen-2-yl)hexane-1,4-dione (2g)

Prepared according to the general procedure with indium homoenolate **1e** (0.096 g, 0.3 mmol), acid chloride (0.095 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 89% isolated yield (0.107 g, 0.45 mmol).

^{1}H NMR (300 MHz, $CDCl_3$): δ 1.10 (t, *J* = 7.32 Hz, 3H), 2.56 (q, *J* = 7.32 Hz, 2H), 2.87 (t, *J* = 6.48 Hz, 2H), 3.39 (t, *J* = 6.13 Hz, 2H), 7.48–7.59 (m, 2H), 7.81–7.86 (m, 2H), 7.92 (d, *J* = 7.77 Hz, 1H), 8.00 (dd, *J* = 1.68, 8.61 Hz, 1H), 8.48 (s, 1H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$): δ 7.8, 32.4, 35.7, 36.0, 123.6, 126.6, 127.6, 128.2, 128.3, 129.4, 129.6, 132.4, 133.9, 135.5, 198.5, 210.0 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{16}H_{17}O_2$: 241.1229, found: 241.1224. FTIR (KBr, neat): *v* 1713, 1680 cm^{-1}.

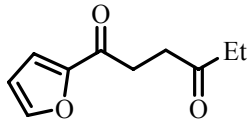


1-(Thiophen-2-yl)hexane-1,4-dione (2h)

Prepared according to the general procedure with indium homoenolate **1e** (0.096 g, 0.3 mmol), acid chloride (0.073 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 90% isolated yield (0.088 g, 0.45 mmol).

^{1}H NMR (500 MHz, $CDCl_3$): δ 1.09 (t, *J* = 7.40 Hz, 3H), 2.54 (q, *J* = 7.42 Hz, 2H), 2.85 (t, *J* = 6.51 Hz, 2H), 3.23 (t, *J* = 6.29 Hz, 2H), 7.13 (dd, *J* = 4.04, 4.80 Hz, 1H), 7.63 (dd, *J* = 0.65, 4.90 Hz, 1H), 7.77 (d, *J* = 3.72 Hz, 1H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 7.8, 32.9, 35.7, 36.0,

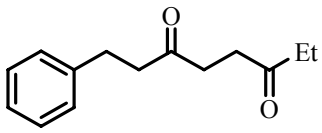
128.1, 131.9, 133.5, 143.7, 191.6, 209.8 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{10}H_{13}O_2S$: 197.0636, found: 197.0642. FTIR (KBr, neat): *v* 1713, 1663 cm^{-1}.



1-(Furan-2-yl)hexane-1,4-dione (2i)

Prepared according to the general procedure with indium homoenolate **1e** (0.096 g, 0.3 mmol), acid chloride (0.065 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 98% isolated yield (0.088 g, 0.49 mmol).

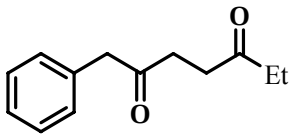
^1H NMR (500 MHz, $CDCl_3$): δ 1.08 (t, *J* = 7.30 Hz, 3H), 2.53 (q, *J* = 7.29 Hz, 2H), 2.84 (t, *J* = 6.59 Hz, 2H), 3.14 (t, *J* = 7.16 Hz, 2H), 6.53–6.54 (m, 1H), 7.21 (d, *J* = 3.55 Hz, 1H), 7.59 (s, 1H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 7.7, 31.9, 35.2, 35.8, 112.1, 116.9, 146.2, 152.3, 187.7, 209.6 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{10}H_{13}O_3$: 181.0865, found: 181.0870. FTIR (KBr, neat): *v* 1713, 1674 cm^{-1}.



1-Phenyloctane-3,6-dione (2j)

Prepared according to the general procedure with indium homoenolate **1e** (0.096 g, 0.3 mmol), acid chloride (0.084 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 69% isolated yield (0.088 g, 0.35 mmol).

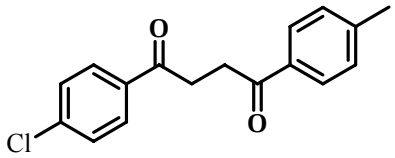
^1H NMR (500 MHz, $CDCl_3$): δ 1.05 (t, *J* = 7.36 Hz, 3H), 2.47 (q, *J* = 7.40 Hz, 2H), 2.66 (s, 4H), 2.79 (t, *J* = 7.90 Hz, 2H), 2.89 (t, *J* = 7.34 Hz, 2H), 7.16–7.19 (m, 3H), 7.25–7.28 (m, 2H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 7.7, 29.6, 35.5, 35.8, 36.1, 44.2, 126.0, 128.2, 128.4, 140.9, 208.5, 209.9 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{14}H_{19}O_2$: 219.1385, found: 219.1390. FTIR (KBr, neat): *v* 1713 cm^{-1}.



1-Phenylheptane-2,5-dione (2k)

Prepared according to the general procedure with indium homoenolate **1e** (0.096 g, 0.3 mmol), acid chloride (0.077 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 64% isolated yield (0.066 g, 0.32 mmol).

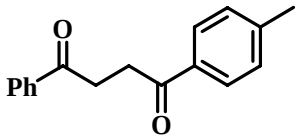
^{1}H NMR (500 MHz, $CDCl_3$): δ 1.05 (t, *J* = 7.35 Hz, 3H), 2.46 (q, *J* = 7.33 Hz, 2H), 2.65 (t, *J* = 6.71 Hz, 2H), 2.73 (t, *J* = 5.64 Hz, 2H), 3.75 (s, 2H), 7.21 (d, *J* = 7.30 Hz, 2H), 7.24–7.27 (m, 1H), 7.33 (t, *J* = 7.30 Hz, 2H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 7.8, 35.6, 35.7, 35.9, 50.1, 127.0, 128.7, 129.5, 134.2, 207.1, 210.0 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{13}H_{17}O_2$: 205.1229, found: 205.1231. FTIR (KBr, neat): *v* 1713 cm^{-1}.



1-(4-Chlorophenyl)-4-*p*-tolylbutane-1,4-dione (2l)[7]

Prepared according to the general procedure with indium homoenolate **1a** (0.146 g, 0.3 mmol), acid chloride (0.077 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 78% isolated yield (0.112 g, 0.39 mmol).

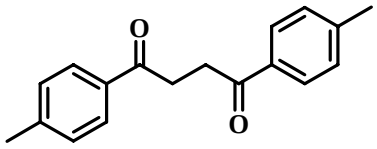
^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.42 (s, 3H), 3.38-3.45 (m, 4H), 7.27 (d, *J* = 8.50 Hz, 2H), 7.45 (d, *J* = 8.57 Hz, 2H), 7.93 (d, *J* = 8.18 Hz, 2H), 7.98 (d, *J* = 8.60 Hz, 2H) ppm. ^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.7, 32.4, 32.5, 128.2, 128.9, 129.3, 129.6, 134.2, 135.1, 139.5, 144.0, 197.6, 198.1 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{17}H_{16}ClO_2$: 287.0839, found: 287.0839. FTIR (KBr, neat): *v* 1674 cm^{-1}.



1-Phenyl-4-*p*-tolylbutane-1,4-dione (2m)[7,8]

Prepared according to the general procedure with indium homoenolate **1b** (0.125 g, 0.3 mmol), acid chloride (0.077 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 70% isolated yield (0.088 g, 0.35 mmol).

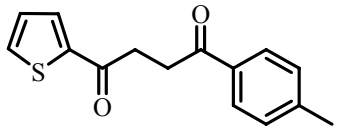
^{1}H NMR (400 MHz, $CDCl_3$): δ 2.42 (s, 3H), 3.45 (s, 4H), 7.28 (d, *J* = 8.09 Hz, 2H), 7.48 (t, *J* = 7.59 Hz, 2H), 7.58 (t, *J* = 7.38 Hz, 1H), 7.94 (d, *J* = 8.21 Hz, 2H), 8.04 (d, *J* = 7.16 Hz, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ 21.7, 32.5, 32.6, 128.1, 128.3, 128.6, 129.3, 133.1, 134.3, 136.8, 144.0, 198.3, 198.8 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{17}H_{17}O_2$: 253.1229, found: 253.1229. FTIR (KBr, neat): *v* 1678 cm^{-1}.



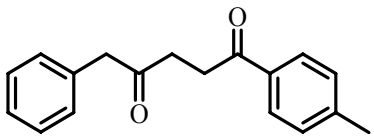
1,4-Dip-tolylbutane-1,4-dione (2n)

Prepared according to the general procedure with indium homoenolate **1c** (0.133 g, 0.3

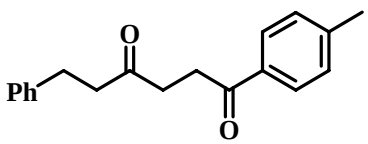
mmol), acid chloride (0.077 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 86% isolated yield (0.115 g, 0.43 mmol).
^{1}H NMR ($CDCl_3$, 300 MHz): δ 2.39 (s, 6H), 3.40 (s, 4H), 7.25 (d, J = 8.04 Hz, 4H), 7.93 (d, J = 8.14 Hz, 4H) ppm. ^{13}C NMR ($CDCl_3$, 75 MHz): δ 21.5, 32.4, 128.2, 129.2, 134.3, 143.8, 198.3 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{18}H_{19}O_2$: 267.1385, found: 267.1392. FTIR (KBr, neat): v 1670 cm^{-1}.



1-(Thiophen-2-yl)-4-*p*-tolylbutane-1,4-dione (2o)
Prepared according to the general procedure with indium homoenolate **1d** (0.129 g, 0.3 mmol), acid chloride (0.077 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 85% isolated yield (0.110 g, 0.43 mmol).
^{1}H NMR ($CDCl_3$, 500 MHz): δ 2.42 (s, 3H), 3.38-3.45 (m, 4H), 7.15 (t, J = 4.45 Hz, 1H), 7.27 (d, J = 8.05 Hz, 2H), 7.64 (d, J = 4.89 Hz, 1H), 7.83 (d, J = 3.79 Hz, 1H), 7.92 (d, J = 8.04 Hz, 2H) ppm. ^{13}C NMR ($CDCl_3$, 125 MHz): δ 21.7, 32.5, 33.2, 128.1, 128.2, 129.3, 132.1, 133.6, 134.2, 143.9, 144.0, 191.8, 198.1 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{15}H_{15}O_2S$: 259.0793, found: 259.0796. FTIR (KBr, neat): v 1674, 1655 cm^{-1}.

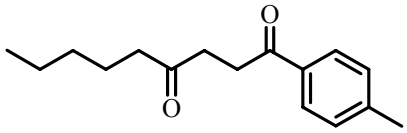


5-Phenyl-1-*p*-tolylpentane-1,4-dione (2p)
Prepared according to the general procedure with indium homoenolate **1f** (0.133 g, 0.3 mmol), acid chloride (0.077 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 79% isolated yield (0.105 g, 0.39 mmol).
^{1}H NMR ($CDCl_3$, 500 MHz): δ 2.39 (s, 3H), 2.87 (t, J = 6.40 Hz, 2H), 3.23 (t, J = 6.36 Hz, 2H), 3.82 (s, 2H), 7.23-7.28 (m, 5H), 7.33 (t, J = 7.44 Hz, 2H), 7.85 (d, J = 8.10 Hz, 2H) ppm. ^{13}C NMR ($CDCl_3$, 125 MHz): δ 21.6, 32.4, 35.7, 50.3, 127.0, 128.1, 128.7, 129.2, 129.5, 134.1, 134.3, 143.9, 198.1, 207.2 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{18}H_{19}O_2$: 267.1385, found: 267.1386. FTIR (KBr, neat): v 1709, 1682 cm^{-1}.

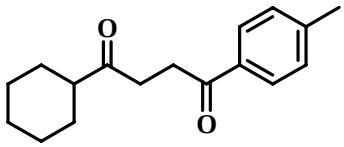


6-Phenyl-1-*p*-tolylhexane-1,4-dione (2q)

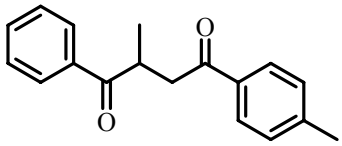
Prepared according to the general procedure with indium homoenolate **1g** (0.142 g, 0.3 mmol), acid chloride (0.077 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 84% isolated yield (0.118 g, 0.42 mmol).
^{1}H NMR (500 MHz, $CDCl_3$): δ 2.40 (s, 3H), 2.83 (t, *J* = 6.39 Hz, 2H), 2.85–2.95 (m, 4H), 3.26 (t, *J* = 6.29 Hz, 2H), 7.19–7.30 (m, 7H), 7.87 (d, *J* = 8.11 Hz, 2H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ 21.7, 29.8, 32.3, 36.4, 44.5, 126.1, 128.2, 128.3, 128.5, 129.3, 134.1, 141.1, 144.0, 198.2, 208.7 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{19}H_{21}O_2$: 281.1542, found: 281.1541. FTIR (KBr, neat): *v* 1709, 1680 cm^{-1}.



1-*p*-Tolylnonane-1,4-dione (2r)
Prepared according to the general procedure with indium homoenolate **1h** (0.121 g, 0.3 mmol), acid chloride (0.077 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 98% isolated yield (0.121 g, 0.49 mmol).
^{1}H NMR ($CDCl_3$, 500 MHz): δ 0.89 (t, *J* = 6.90 Hz, 3H), 1.26-1.35 (m, 4H), 1.59-1.65 (m, 2H), 2.40 (s, 3H), 2.52 (t, *J* = 7.46 Hz, 2H), 2.84 (t, *J* = 6.40 Hz, 2H), 3.25 (t, *J* = 6.20 Hz, 2H), 7.25 (d, *J* = 8.02 Hz, 2H), 7.88 (d, *J* = 8.15 Hz, 2H) ppm. ^{13}C NMR ($CDCl_3$, 125 MHz): δ 13.9, 21.6, 22.4, 23.5, 31.4, 32.2, 36.2, 42.9, 128.1, 129.2, 134.2, 143.8, 198.3, 209.9 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{16}H_{23}O_2$: 247.1698, found: 247.1696. FTIR (KBr, neat): *v* 1709, 1680 cm^{-1}.



1-Cyclohexyl-4-*p*-tolylbutane-1,4-dione (2s)
Prepared according to the general procedure with indium homoenolate **1i** (0.129 g, 0.3 mmol), acid chloride (0.077 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 70% isolated yield (0.091 g, 0.35 mmol).
^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.19-1.42 (m, 5H), 1.65-1.69 (m, 1H), 1.77-1.81 (m, 2H), 1.91-1.93 (m, 2H), 2.40 (s, 3H), 2.46 (tt, *J* = 11.09, 3.25 Hz, 1H), 2.87 (t, *J* = 6.45 Hz, 2H), 3.24 (t, *J* = 6.16 Hz, 2H), 7.24 (d, *J* = 8.00 Hz, 2H), 7.88 (d, *J* = 8.16 Hz, 2H) ppm. ^{13}C NMR ($CDCl_3$, 125 MHz): δ 21.6, 25.6, 25.8, 28.5, 32.2, 34.2, 50.9, 128.1, 129.2, 134.2, 143.8, 198.3, 212.7 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{17}H_{23}O_2$: 259.1698, found: 259.1702. FTIR (KBr, neat): *v* 1703, 1686 cm^{-1}.



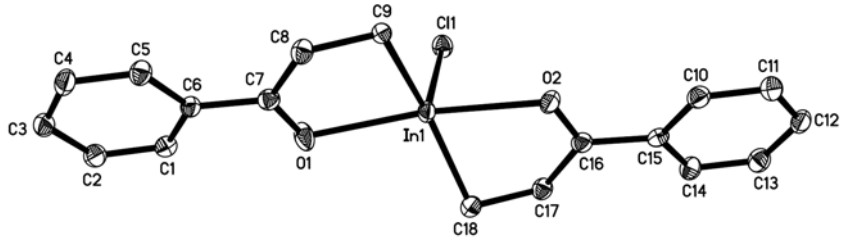
2-Methyl-1-phenyl-4-*p*-tolylbutane-1,4-dione (2t)

Prepared according to the general procedure with indium homoenolate **1j** (0.129 g, 0.3 mmol), acid chloride (0.077 g, 0.5 mmol), $PdCl_2(PPh_3)_2$ (0.018 g, 0.025 mmol) in THF (3 mL) to afford the title compound in 85% isolated yield (0.113 g, 0.42 mmol).

^{1}H NMR ($CDCl_3$, 500 MHz): δ 1.28 (d, *J* = 7.14 Hz, 3H), 2.40 (s, 3H), 3.10 (dd, *J* = 17.86, 4.94 Hz, 1H), 3.69 (dd, *J* = 17.89, 8.35 Hz, 1H), 4.14-4.21 (m, 1H), 7.25 (d, *J* = 8.15 Hz, 2H), 7.48 (t, *J* = 7.80 Hz, 2H), 7.57 (t, *J* = 7.36 Hz, 1H), 7.88 (d, *J* = 8.21 Hz, 2H), 8.05 (d, *J* = 7.25 Hz, 2H) ppm. ^{13}C NMR ($CDCl_3$, 125 MHz): δ 17.9, 21.6, 36.3, 42.2, 128.2, 128.5, 128.6, 129.2, 132.9, 134.2, 136.1, 144.0, 198.1, 203.5 ppm. HRMS (ESI, m/z): $[M+H]^+$, calcd. for $C_{18}H_{19}O_2$: 267.1385, found: 267.1393. FTIR (KBr, neat): *v* 1674 cm^{-1}.

X-Ray crystal structure information

Table 1. Crystal data and structure refinement for ltp170s.



Identification code	ltp170s	
Empirical formula	C18 H18 Cl In O2	
Formula weight	416.59	
Temperature	103(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	Cc	
Unit cell dimensions	a = 19.6727(4) Å	α= 90°.
	b = 7.74200(10) Å	β= 91.7910(10)°.
	c = 10.7323(2) Å	γ = 90°.
Volume	1633.80(5) Å^3	
Z	4	
Density (calculated)	1.694 Mg/m^3	

Absorption coefficient	1.614 mm^{-1}
F(000)	832
Crystal size	0.38 x 0.14 x 0.10 mm^3
Theta range for data collection	2.07 to 34.13°.
Index ranges	-30<=h<=30, -12<=k<=12, -14<=l<=16
Reflections collected	12912
Independent reflections	6193 [R(int) = 0.0234]
Completeness to theta = 34.13°	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.8553 and 0.5791
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6193 / 95 / 249
Goodness-of-fit on F^2	1.018
Final R indices [I>2sigma(I)]	R1 = 0.0268, wR2 = 0.0621
R indices (all data)	R1 = 0.0300, wR2 = 0.0637
Absolute structure parameter	0.250(15)
Largest diff. peak and hole	0.776 and -0.356 e.$Å^{-3}$

Table 2. Atomic coordinates (x10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ltp170s. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
In(1)	1494(1)	9788(1)	6661(1)	16(1)
C(9)	1208(3)	7607(6)	5535(7)	13(1)
C(18)	1839(4)	12383(9)	7129(10)	17(2)
Cl(1)	1539(3)	8490(3)	8837(6)	20(1)
O(1)	275(3)	10215(10)	6368(8)	32(2)
O(2)	2717(2)	9664(6)	6567(4)	18(1)
In(1A)	1545(1)	10200(1)	6083(1)	19(1)
C(9A)	1199(10)	7682(19)	5620(30)	34(5)
C(18A)	1812(9)	12280(20)	7270(30)	13(3)
Cl(1A)	1464(7)	11479(7)	3906(15)	20(1)
O(1A)	321(6)	10000(20)	6493(13)	12(2)
O(2A)	2774(5)	9901(16)	6268(8)	18(2)
C(1)	-1101(1)	10245(3)	6055(2)	18(1)
C(2)	-1798(1)	10346(3)	5803(2)	21(1)

C(3)	-2094(1)	9316(3)	4874(2)	21(1)
C(4)	-1700(1)	8184(3)	4203(2)	22(1)
C(5)	-1001(1)	8092(3)	4449(2)	19(1)
C(6)	-698(1)	9134(2)	5369(2)	16(1)
C(7)	45(1)	9101(2)	5645(2)	17(1)
C(8)	497(1)	7865(3)	4961(2)	19(1)
C(10)	4137(1)	9744(3)	6717(2)	19(1)
C(11)	4829(1)	9667(3)	6955(2)	21(1)
C(12)	5132(1)	10718(3)	7869(2)	21(1)
C(13)	4734(1)	11839(3)	8538(2)	22(1)
C(14)	4038(1)	11921(3)	8318(2)	19(1)
C(15)	3732(1)	10862(2)	7400(2)	16(1)
C(16)	2985(1)	10852(3)	7158(2)	17(1)
C(17)	2540(1)	12168(2)	7771(2)	18(1)

Table 3. Bond lengths [Å] and angles [°] for ltp170s.

In(1)-C(9)	2.141(6)
In(1)-C(18)	2.174(6)
In(1)-O(2)	2.413(5)
In(1)-O(1)	2.431(7)
In(1)-Cl(1)	2.542(6)
C(9)-C(8)	1.525(5)
C(9)-H(9A)	0.9900
C(9)-H(9B)	0.9900
C(18)-C(17)	1.532(6)
C(18)-H(18A)	0.9900
C(18)-H(18B)	0.9900
O(1)-C(7)	1.236(6)
O(2)-C(16)	1.227(5)
In(1A)-C(18A)	2.111(15)
In(1A)-C(9A)	2.118(14)
In(1A)-O(2A)	2.431(11)
In(1A)-O(1A)	2.467(11)
In(1A)-Cl(1A)	2.538(15)
C(9A)-C(8)	1.542(14)

C(9A)-H(9A1)	0.9900
C(9A)-H(9A2)	0.9900
C(18A)-C(17)	1.516(14)
C(18A)-H(18C)	0.9900
C(18A)-H(18D)	0.9900
O(1A)-C(7)	1.254(12)
O(2A)-C(16)	1.266(10)
C(1)-C(2)	1.393(3)
C(1)-C(6)	1.395(3)
C(1)-H(1)	0.9500
C(2)-C(3)	1.390(3)
C(2)-H(2)	0.9500
C(3)-C(4)	1.387(3)
C(3)-H(3)	0.9500
C(4)-C(5)	1.394(3)
C(4)-H(4)	0.9500
C(5)-C(6)	1.394(2)
C(5)-H(5)	0.9500
C(6)-C(7)	1.483(3)
C(7)-C(8)	1.512(3)
C(8)-H(8A)	0.9900
C(8)-H(8B)	0.9900
C(10)-C(11)	1.380(3)
C(10)-C(15)	1.399(3)
C(10)-H(10)	0.9500
C(11)-C(12)	1.394(3)
C(11)-H(11)	0.9500
C(12)-C(13)	1.385(3)
C(12)-H(12)	0.9500
C(13)-C(14)	1.382(3)
C(13)-H(13)	0.9639
C(14)-C(15)	1.403(3)
C(14)-H(14)	0.9500
C(15)-C(16)	1.484(3)
C(16)-C(17)	1.508(3)
C(17)-H(17A)	0.9900
C(17)-H(17B)	0.9900

C(9)-In(1)-C(18)	159.0(3)
C(9)-In(1)-O(2)	101.0(2)
C(18)-In(1)-O(2)	75.1(2)
C(9)-In(1)-O(1)	78.1(2)
C(18)-In(1)-O(1)	101.7(2)
O(2)-In(1)-O(1)	168.7(3)
C(9)-In(1)-Cl(1)	102.0(2)
C(18)-In(1)-Cl(1)	98.7(3)
O(2)-In(1)-Cl(1)	90.96(16)
O(1)-In(1)-Cl(1)	100.3(2)
C(8)-C(9)-In(1)	110.3(3)
C(8)-C(9)-H(9A)	109.6
In(1)-C(9)-H(9A)	109.6
C(8)-C(9)-H(9B)	109.6
In(1)-C(9)-H(9B)	109.6
H(9A)-C(9)-H(9B)	108.1
C(17)-C(18)-In(1)	105.9(4)
C(17)-C(18)-H(18A)	110.6
In(1)-C(18)-H(18A)	110.6
C(17)-C(18)-H(18B)	110.6
In(1)-C(18)-H(18B)	110.6
H(18A)-C(18)-H(18B)	108.7
C(7)-O(1)-In(1)	109.1(4)
C(16)-O(2)-In(1)	111.2(3)
C(18A)-In(1A)-C(9A)	156.2(11)
C(18A)-In(1A)-O(2A)	78.2(6)
C(9A)-In(1A)-O(2A)	104.0(6)
C(18A)-In(1A)-O(1A)	99.6(7)
C(9A)-In(1A)-O(1A)	71.0(8)
O(2A)-In(1A)-O(1A)	162.5(5)
C(18A)-In(1A)-Cl(1A)	105.5(8)
C(9A)-In(1A)-Cl(1A)	97.7(7)
O(2A)-In(1A)-Cl(1A)	98.4(4)
O(1A)-In(1A)-Cl(1A)	98.9(5)
C(8)-C(9A)-In(1A)	107.4(8)
C(8)-C(9A)-H(9A1)	110.2
In(1A)-C(9A)-H(9A1)	110.2
C(8)-C(9A)-H(9A2)	110.2

In(1A)-C(9A)-H(9A2)	110.2
H(9A1)-C(9A)-H(9A2)	108.5
C(17)-C(18A)-In(1A)	112.6(10)
C(17)-C(18A)-H(18C)	109.1
In(1A)-C(18A)-H(18C)	109.1
C(17)-C(18A)-H(18D)	109.1
In(1A)-C(18A)-H(18D)	109.1
H(18C)-C(18A)-H(18D)	107.8
C(7)-O(1A)-In(1A)	107.9(7)
C(16)-O(2A)-In(1A)	107.9(7)
C(2)-C(1)-C(6)	120.29(18)
C(2)-C(1)-H(1)	119.9
C(6)-C(1)-H(1)	119.9
C(3)-C(2)-C(1)	119.70(19)
C(3)-C(2)-H(2)	120.1
C(1)-C(2)-H(2)	120.1
C(4)-C(3)-C(2)	120.38(19)
C(4)-C(3)-H(3)	119.8
C(2)-C(3)-H(3)	119.8
C(3)-C(4)-C(5)	119.90(18)
C(3)-C(4)-H(4)	120.0
C(5)-C(4)-H(4)	120.0
C(6)-C(5)-C(4)	120.17(19)
C(6)-C(5)-H(5)	119.9
C(4)-C(5)-H(5)	119.9
C(5)-C(6)-C(1)	119.54(18)
C(5)-C(6)-C(7)	121.94(17)
C(1)-C(6)-C(7)	118.53(16)
O(1)-C(7)-O(1A)	10.6(12)
O(1)-C(7)-C(6)	116.9(4)
O(1A)-C(7)-C(6)	122.5(6)
O(1)-C(7)-C(8)	122.5(4)
O(1A)-C(7)-C(8)	117.1(6)
C(6)-C(7)-C(8)	120.27(16)
C(7)-C(8)-C(9)	115.6(3)
C(7)-C(8)-C(9A)	111.5(9)
C(9)-C(8)-C(9A)	4.2(13)
C(7)-C(8)-H(8A)	108.4

C(9)-C(8)-H(8A)	108.4
C(9A)-C(8)-H(8A)	111.0
C(7)-C(8)-H(8B)	108.4
C(9)-C(8)-H(8B)	108.4
C(9A)-C(8)-H(8B)	110.0
H(8A)-C(8)-H(8B)	107.4
C(11)-C(10)-C(15)	120.36(19)
C(11)-C(10)-H(10)	119.8
C(15)-C(10)-H(10)	119.8
C(10)-C(11)-C(12)	120.2(2)
C(10)-C(11)-H(11)	119.9
C(12)-C(11)-H(11)	119.9
C(13)-C(12)-C(11)	119.63(19)
C(13)-C(12)-H(12)	120.2
C(11)-C(12)-H(12)	120.2
C(14)-C(13)-C(12)	120.87(18)
C(14)-C(13)-H(13)	121.5
C(12)-C(13)-H(13)	117.5
C(13)-C(14)-C(15)	119.64(19)
C(13)-C(14)-H(14)	120.2
C(15)-C(14)-H(14)	120.2
C(10)-C(15)-C(14)	119.31(19)
C(10)-C(15)-C(16)	118.82(17)
C(14)-C(15)-C(16)	121.84(18)
O(2)-C(16)-O(2A)	17.9(5)
O(2)-C(16)-C(15)	120.1(3)
O(2A)-C(16)-C(15)	115.8(5)
O(2)-C(16)-C(17)	119.1(3)
O(2A)-C(16)-C(17)	122.8(6)
C(15)-C(16)-C(17)	120.34(16)
C(16)-C(17)-C(18A)	116.1(8)
C(16)-C(17)-C(18)	113.8(4)
C(18A)-C(17)-C(18)	6.9(16)
C(16)-C(17)-H(17A)	108.8
C(18A)-C(17)-H(17A)	112.9
C(18)-C(17)-H(17A)	108.8
C(16)-C(17)-H(17B)	108.8
C(18A)-C(17)-H(17B)	102.0

C(18)-C(17)-H(17B)	108.8
H(17A)-C(17)-H(17B)	107.7

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2x10^3$) for ltp170s. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
In(1)	12(1)	17(1)	18(1)	-2(1)	-2(1)	-1(1)
C(9)	15(2)	15(2)	10(2)	0(1)	-1(2)	0(2)
C(18)	18(2)	19(2)	15(3)	1(2)	-1(2)	-3(2)
Cl(1)	18(1)	23(1)	18(1)	2(1)	-1(1)	0(1)
O(1)	21(2)	26(3)	48(3)	-20(3)	-16(2)	7(2)
O(2)	15(1)	18(2)	22(2)	-4(1)	3(1)	-3(1)
In(1A)	17(1)	20(1)	20(1)	-2(1)	-2(1)	-1(1)
C(9A)	26(8)	38(9)	37(9)	-12(6)	9(6)	0(7)
C(18A)	19(5)	13(5)	8(5)	4(3)	5(3)	1(4)
Cl(1A)	18(1)	23(1)	18(1)	2(1)	-1(1)	0(1)
O(1A)	13(4)	9(3)	12(3)	0(2)	-1(3)	5(3)
O(2A)	16(3)	27(4)	13(4)	-6(3)	4(3)	-12(2)
C(1)	19(1)	17(1)	19(1)	-1(1)	1(1)	-1(1)
C(2)	19(1)	20(1)	23(1)	-1(1)	2(1)	0(1)
C(3)	17(1)	21(1)	23(1)	4(1)	-1(1)	-2(1)
C(4)	18(1)	24(1)	22(1)	-2(1)	-4(1)	-4(1)
C(5)	18(1)	21(1)	18(1)	-3(1)	0(1)	-1(1)
C(6)	16(1)	16(1)	16(1)	1(1)	-2(1)	-1(1)
C(7)	19(1)	16(1)	17(1)	0(1)	0(1)	-1(1)
C(8)	17(1)	21(1)	18(1)	-4(1)	1(1)	-2(1)
C(10)	20(1)	18(1)	21(1)	-1(1)	1(1)	-2(1)
C(11)	19(1)	22(1)	24(1)	0(1)	3(1)	0(1)
C(12)	16(1)	22(1)	24(1)	3(1)	0(1)	-4(1)
C(13)	21(1)	23(1)	21(1)	-1(1)	-2(1)	-4(1)
C(14)	19(1)	22(1)	18(1)	-2(1)	-1(1)	-3(1)
C(15)	18(1)	14(1)	16(1)	2(1)	2(1)	-2(1)

C(16)	18(1)	16(1)	18(1)	1(1)	-2(1)	-2(1)
C(17)	18(1)	18(1)	18(1)	-3(1)	1(1)	-2(1)

Table 5. Hydrogen coordinates ($x10^4$) and isotropic displacement parameters ($Å^2 x\ 10^3$) for ltp170s.

	x	y	z	U(eq)
H(9A)	1538	7457	4865	16
H(9B)	1217	6550	6054	16
H(18A)	1872	13095	6366	20
H(18B)	1520	12948	7696	20
H(9A1)	1522	7109	5068	40
H(9A2)	1163	6977	6388	40
H(18C)	1749	13378	6810	16
H(18D)	1502	12295	7983	16
H(1)	-898	10936	6697	22
H(2)	-2071	11114	6264	25
H(3)	-2570	9388	4697	25
H(4)	-1905	7473	3576	26
H(5)	-730	7318	3990	23
H(8A)	541	8290	4097	22
H(8B)	268	6727	4912	22
H(10)	3934	9035	6086	23
H(11)	5101	8895	6495	26
H(13)	4963	12607	9120	26
H(12)	5609	10665	8033	25
H(14)	3770	12690	8786	23
H(17A)	2776	13298	7777	22
H(17B)	2479	11822	8648	22

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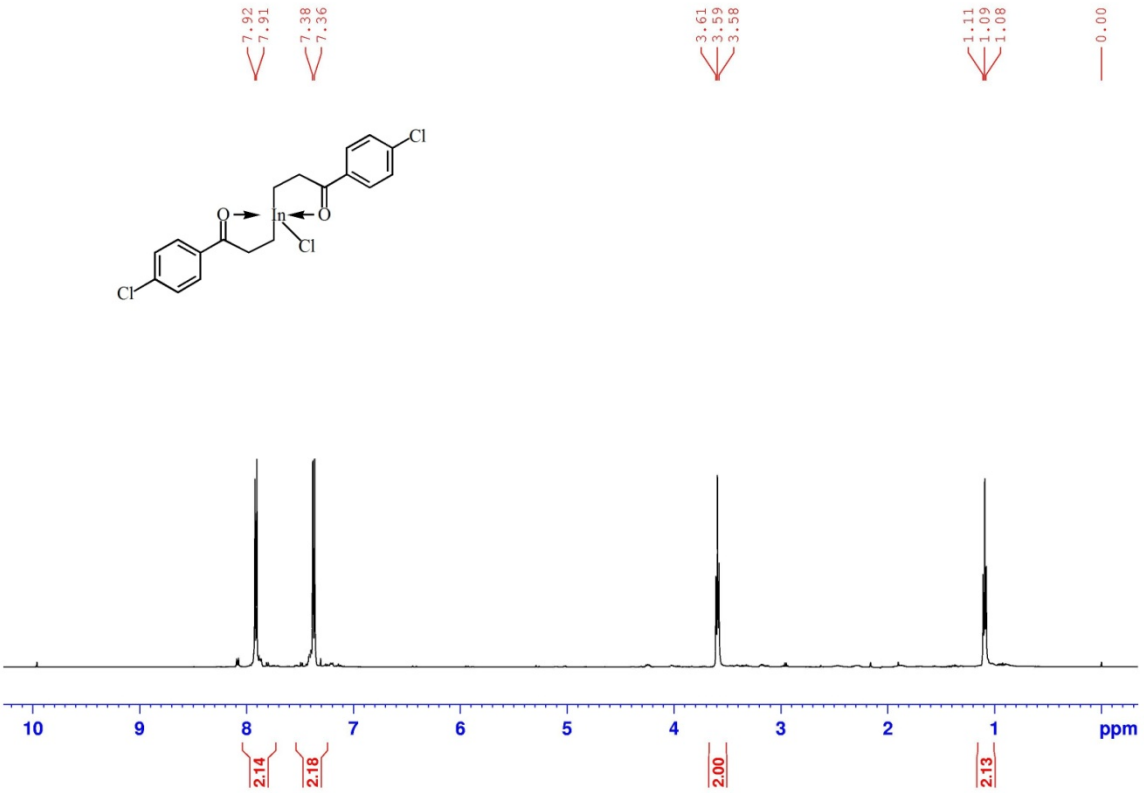
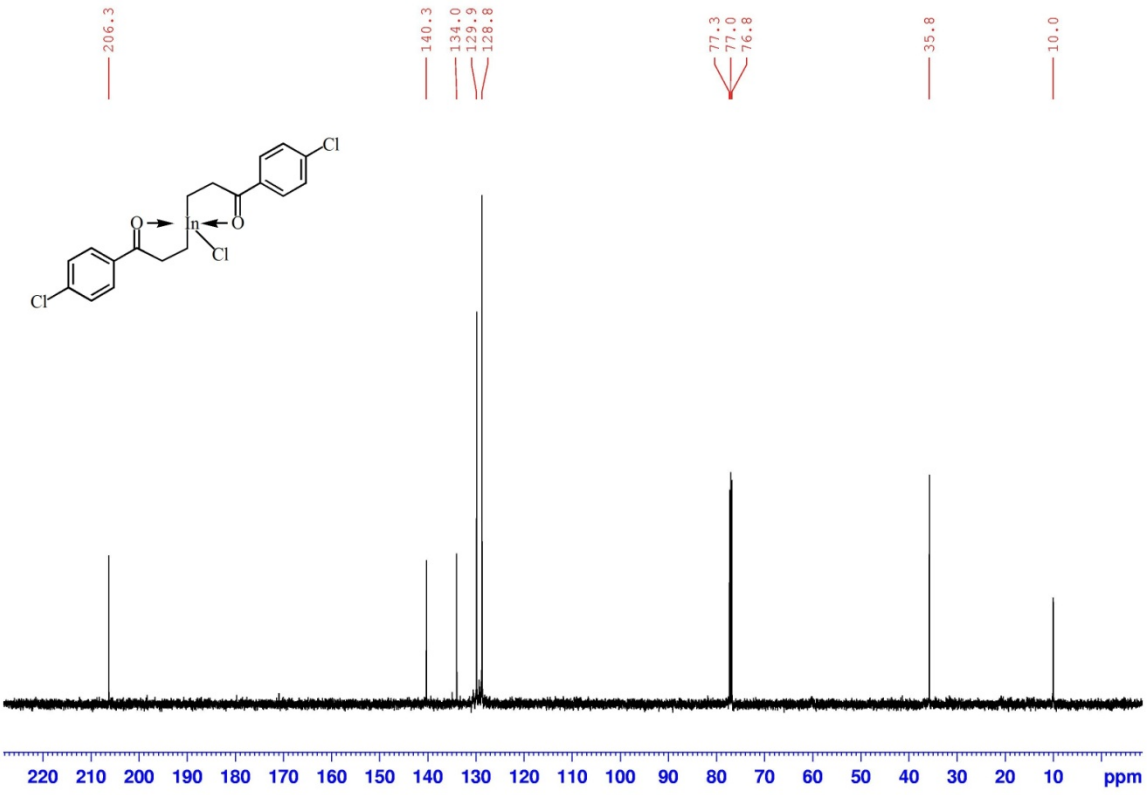
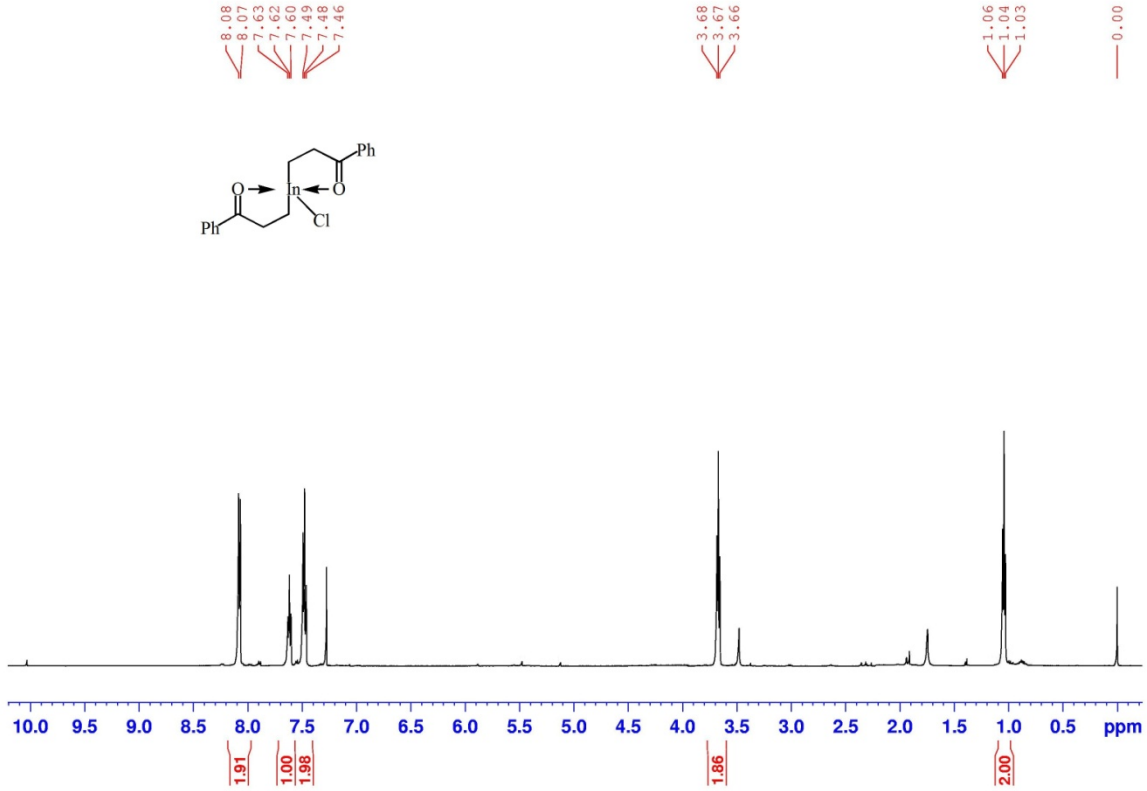
Copies of ^{1}H NMR and ^{13}C NMR



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1.04
1.03
0.00
Ph
O
In
O
Cl
Ph
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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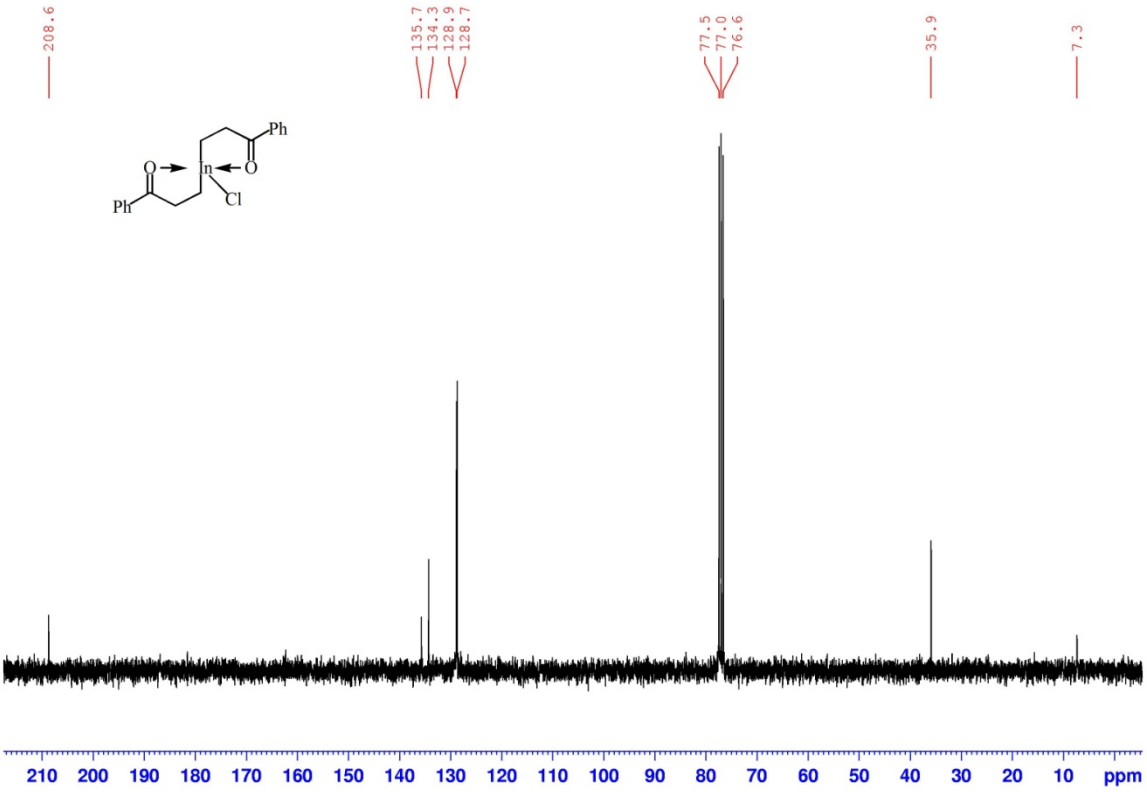
1.86
2.00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76.6
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7.3
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O
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Cl
Ph
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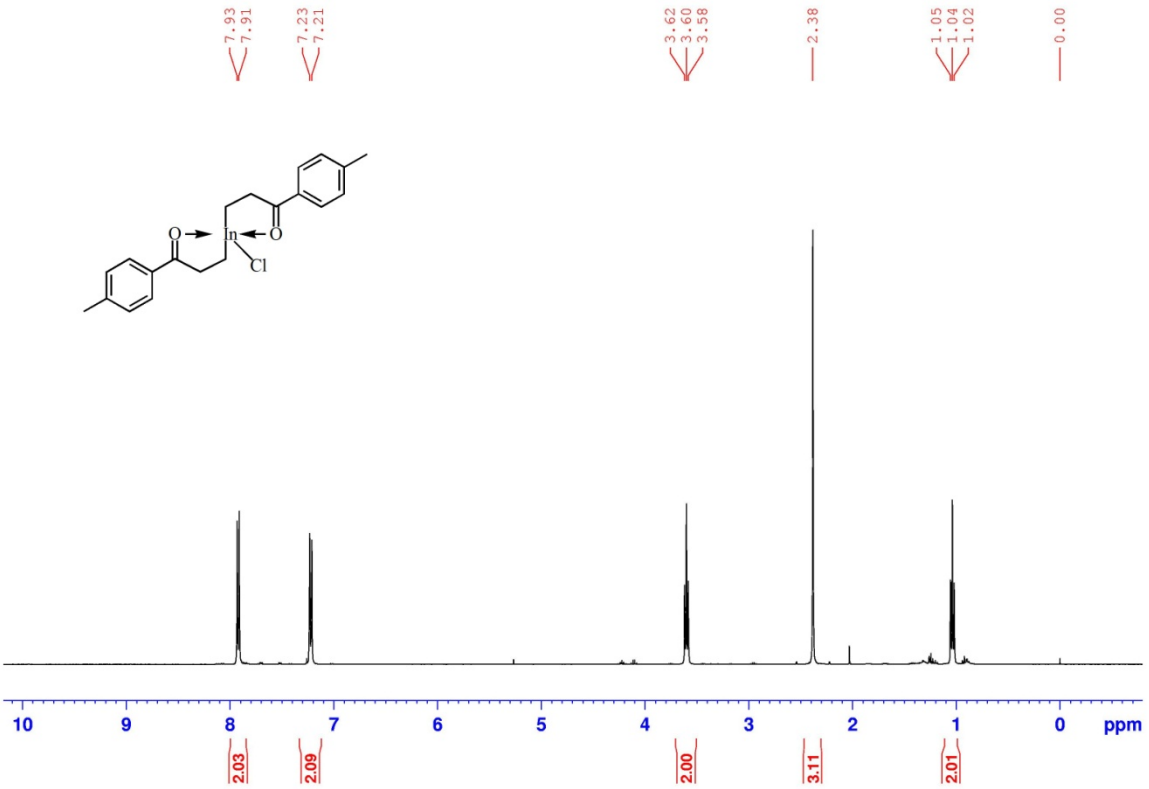
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In
O
Cl
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4
3
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0
ppm
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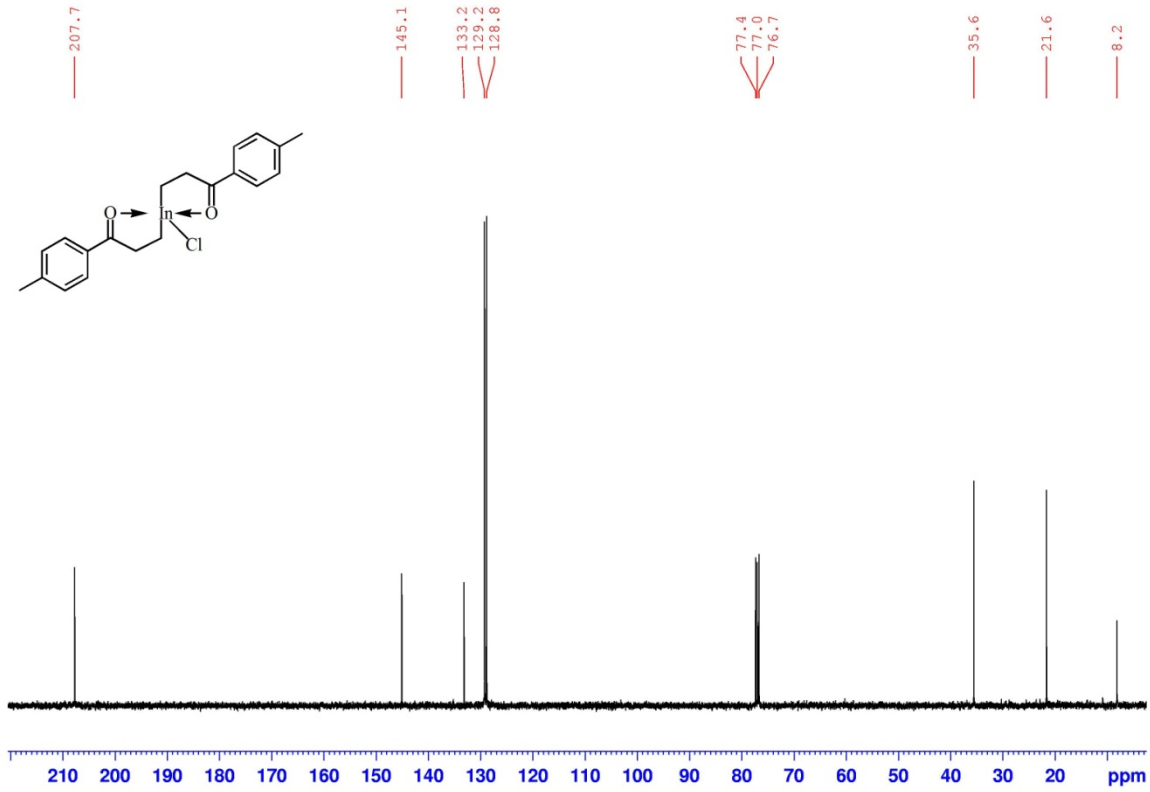
2.00
3.11
2.01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O
Cl
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130
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40
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20
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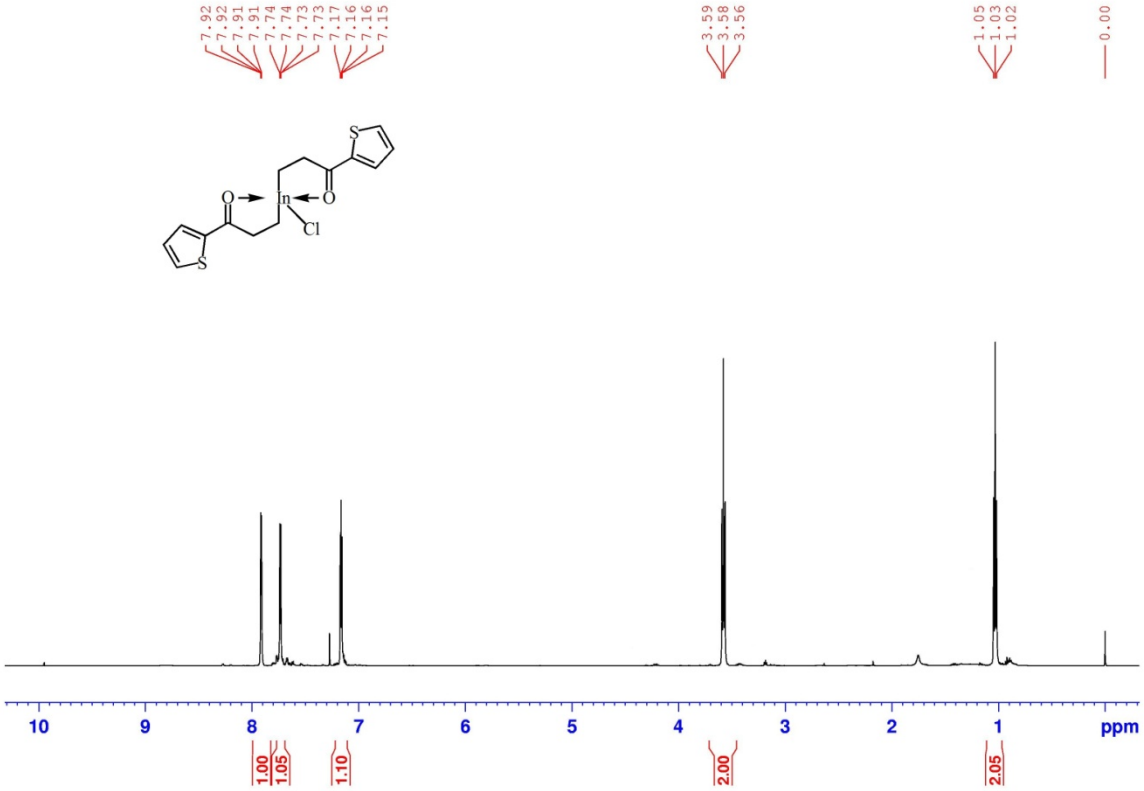
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3.58
3.56
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1.03
1.02
0.00
In
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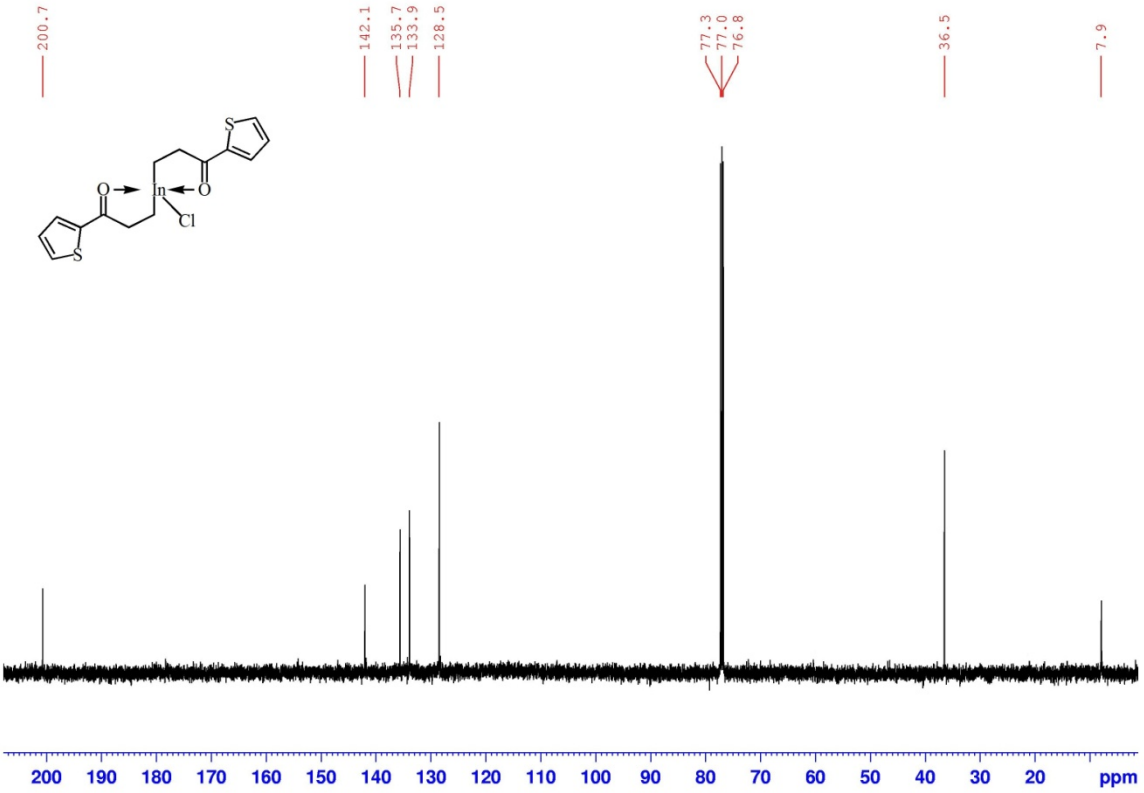
2.05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130
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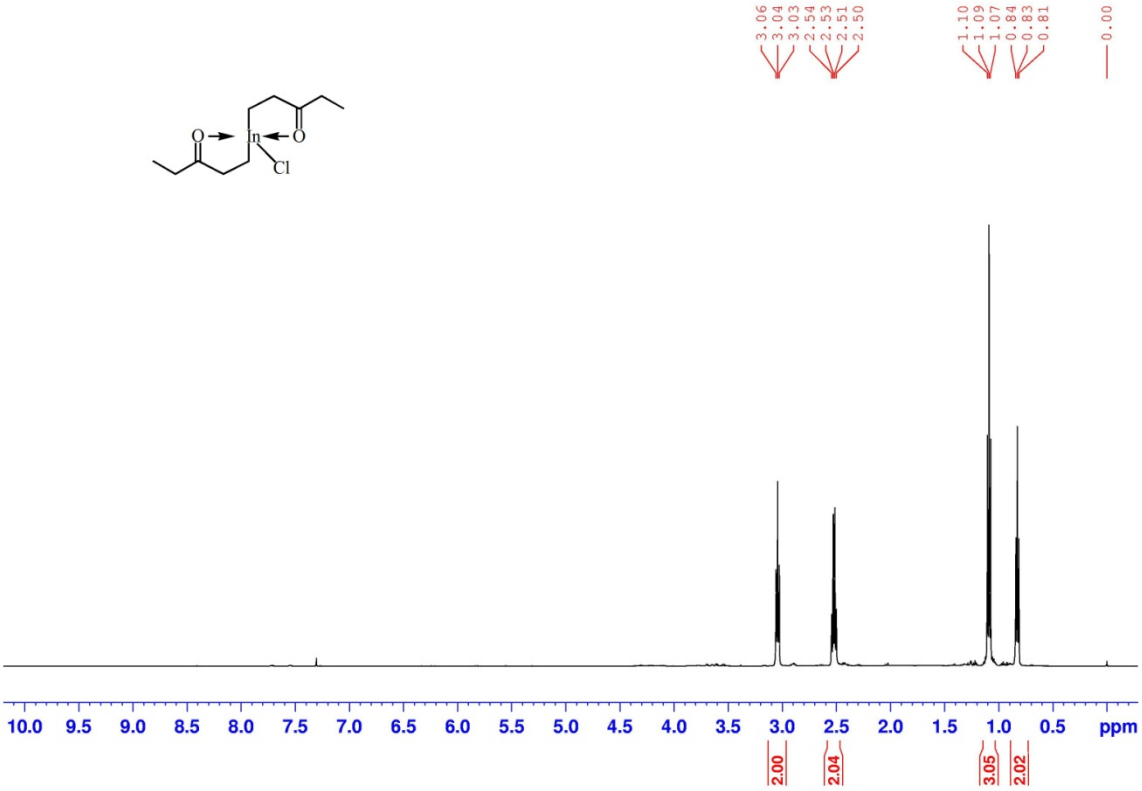
50
40
30
20
ppm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0.00
In
Cl
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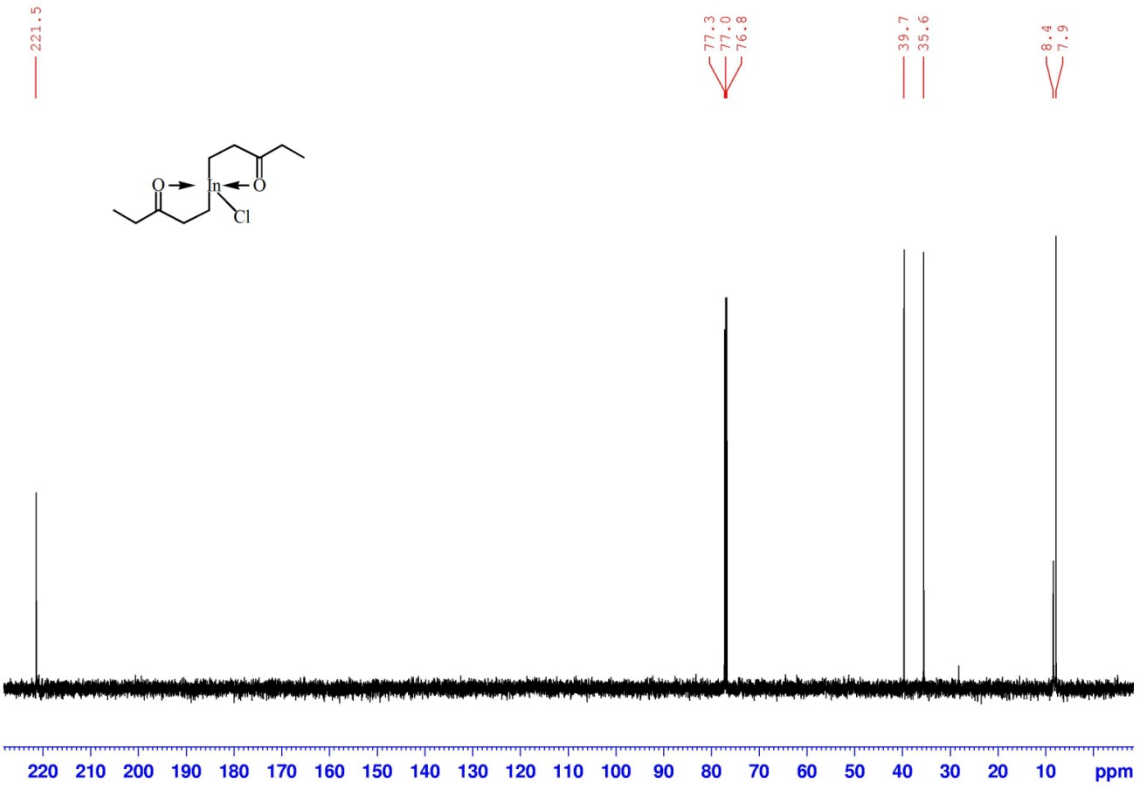
2.04
3.05
2.02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76.8
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8.4
7.9
In
Cl
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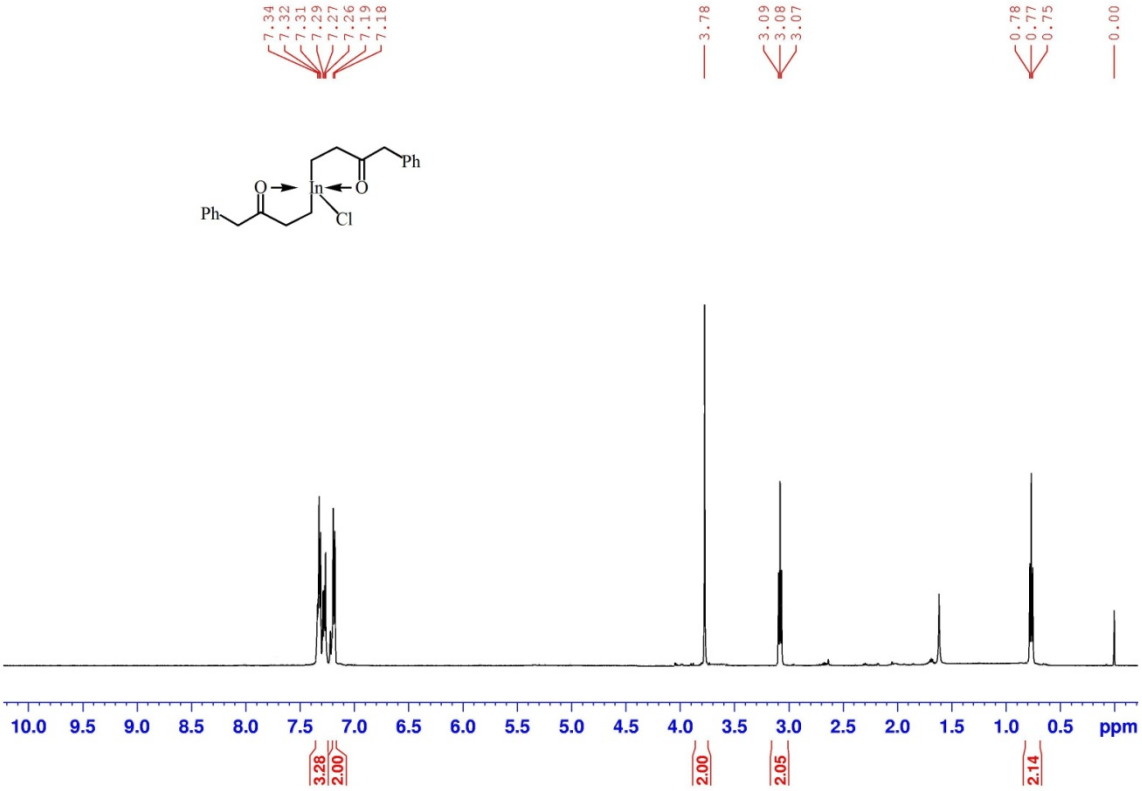
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In
O
Ph
Cl
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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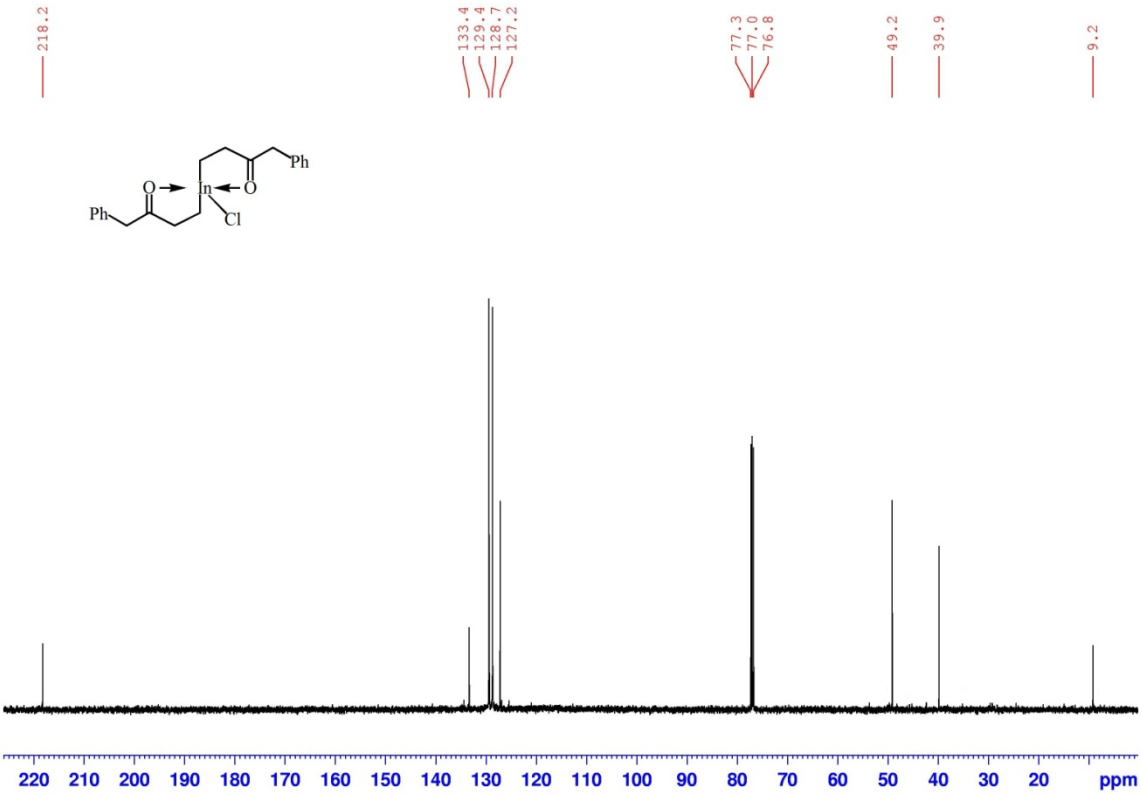
2.14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Ph
Cl
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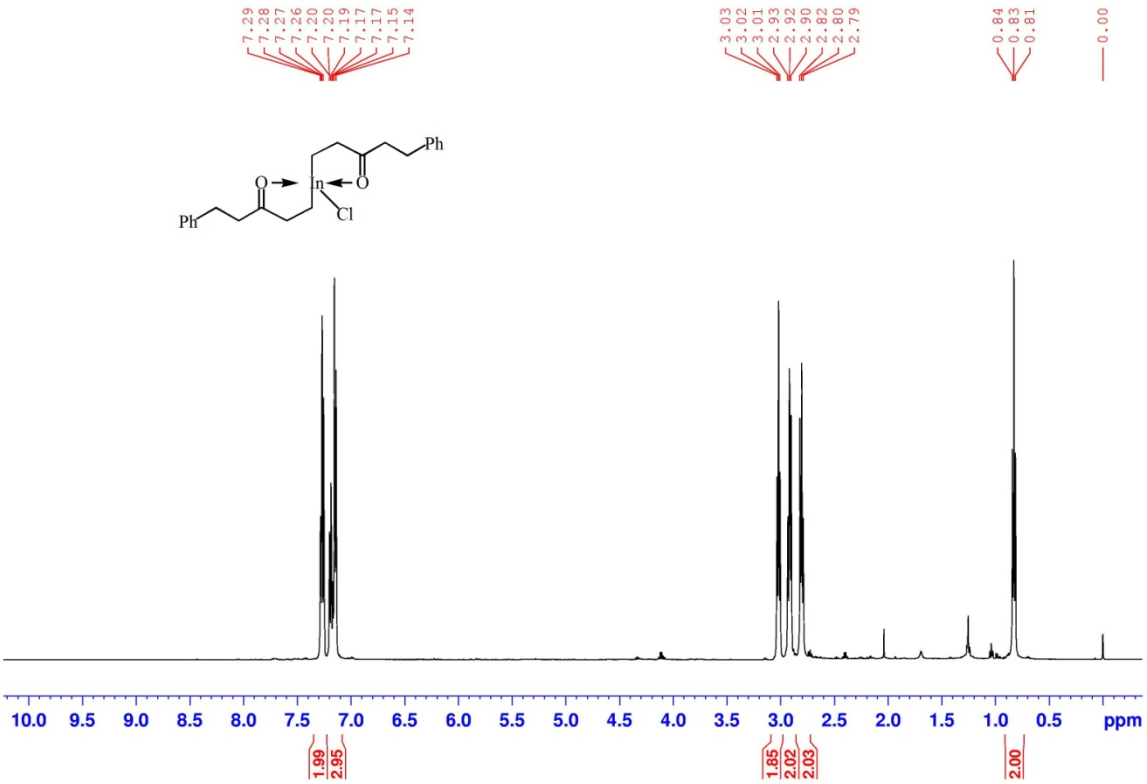
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In
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Cl
Ph
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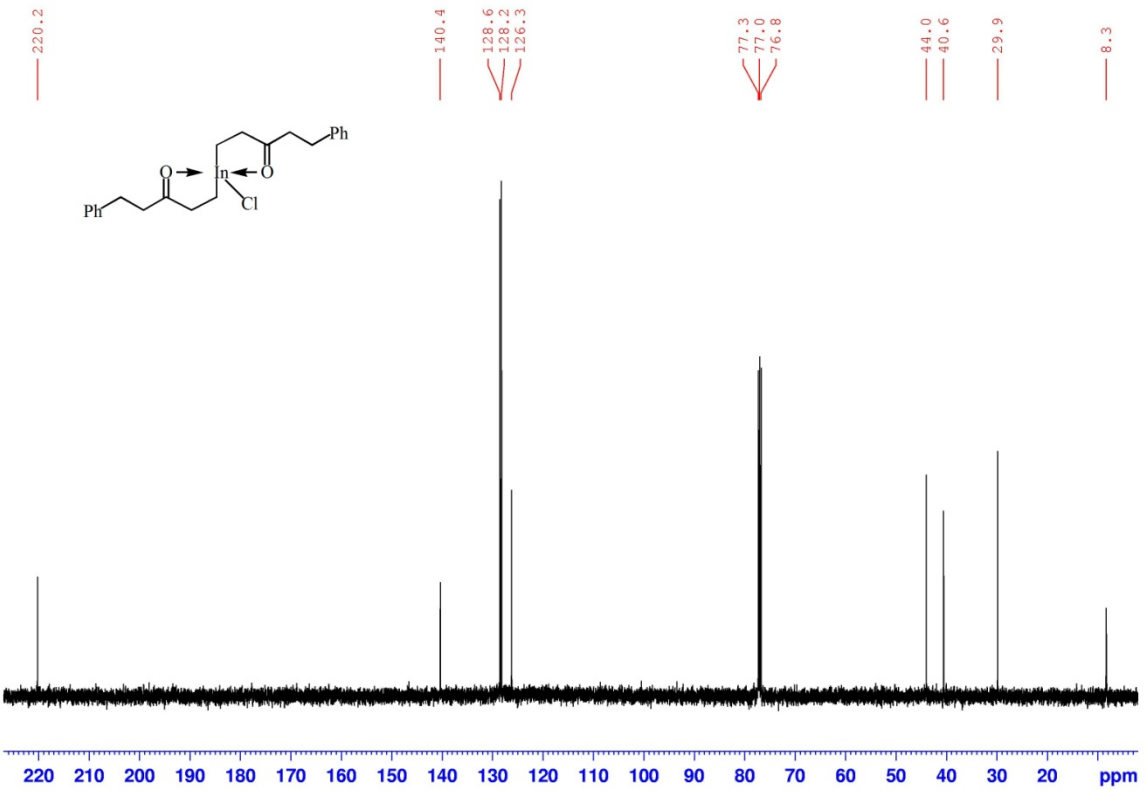
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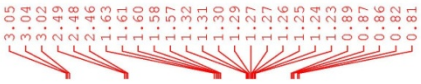
In
O
Cl
Ph
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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1.30
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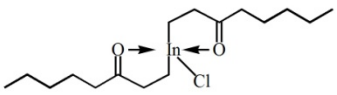
0.81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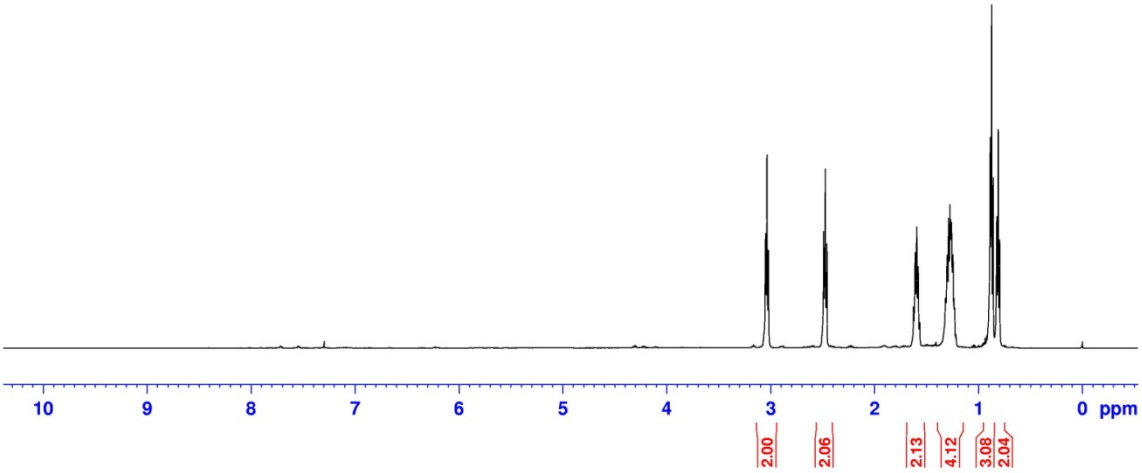
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2.13
4.12
3.08
2.04


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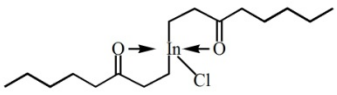
22.3
13.8
8.4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O
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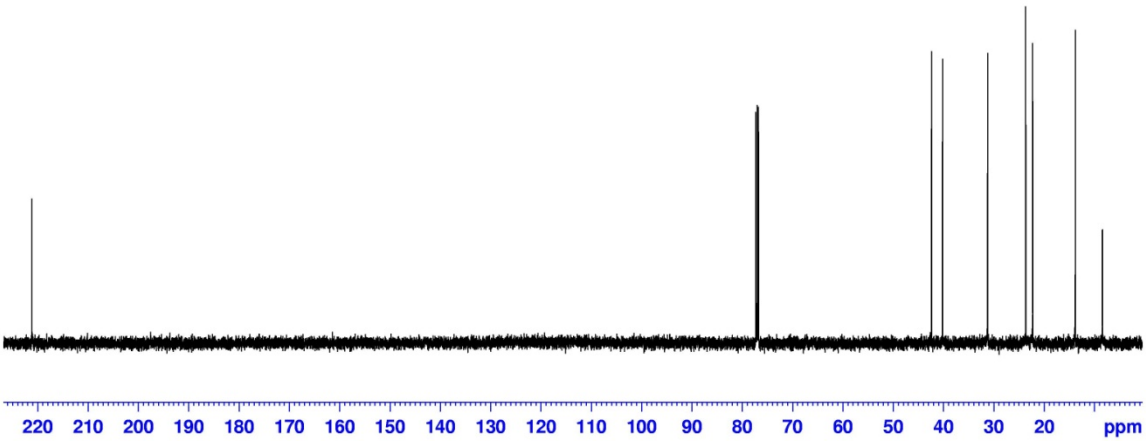
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50
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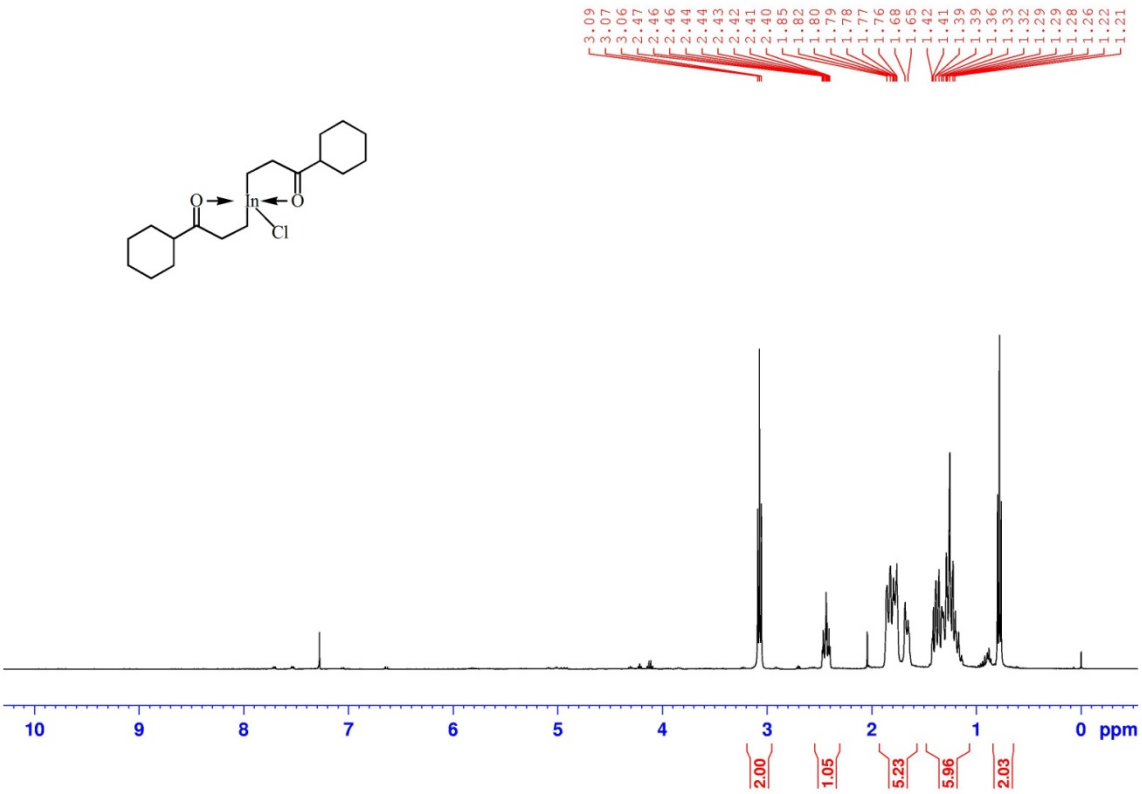
30
20
ppm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1.41
1.39
1.39
1.36
1.33
1.32
1.29
1.29
1.28
1.26
1.22
1.21
O
In
O
Cl
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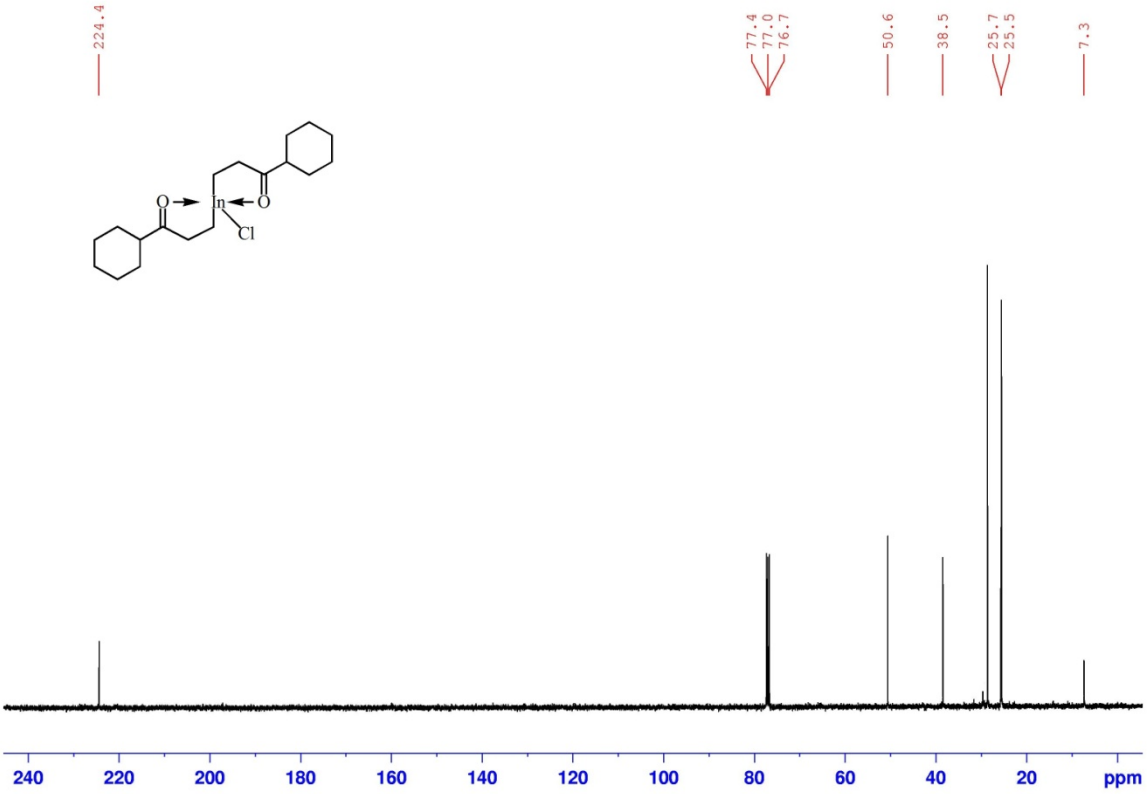
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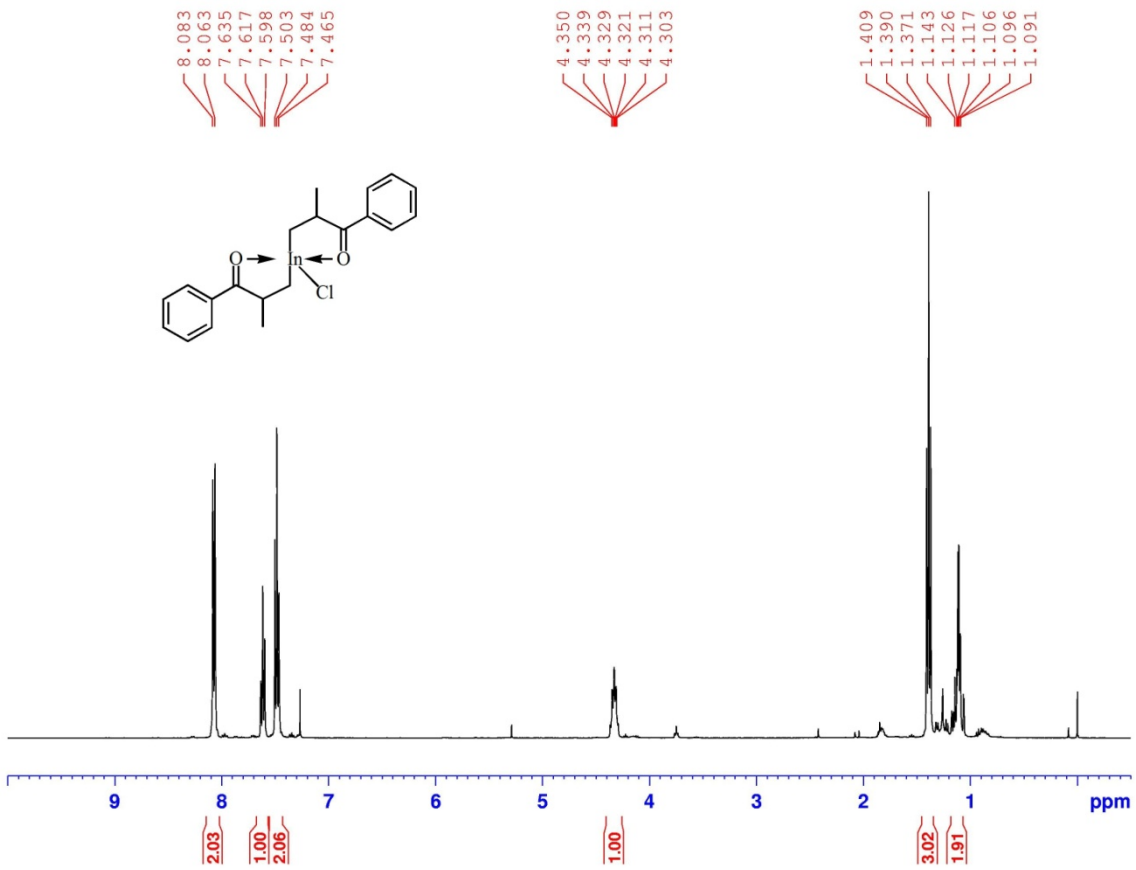
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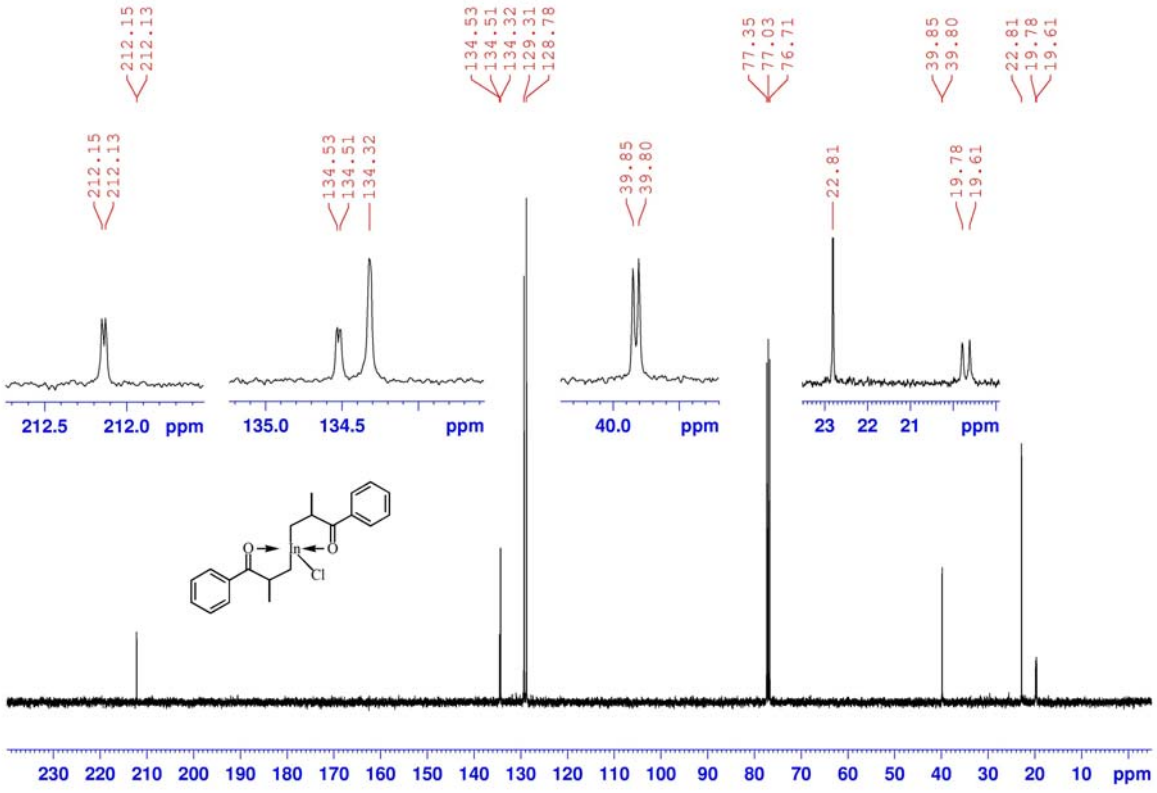
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19.61
212.5
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ppm
135.0
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ppm
40.0
ppm
23
22
21
ppm
In
Cl
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220
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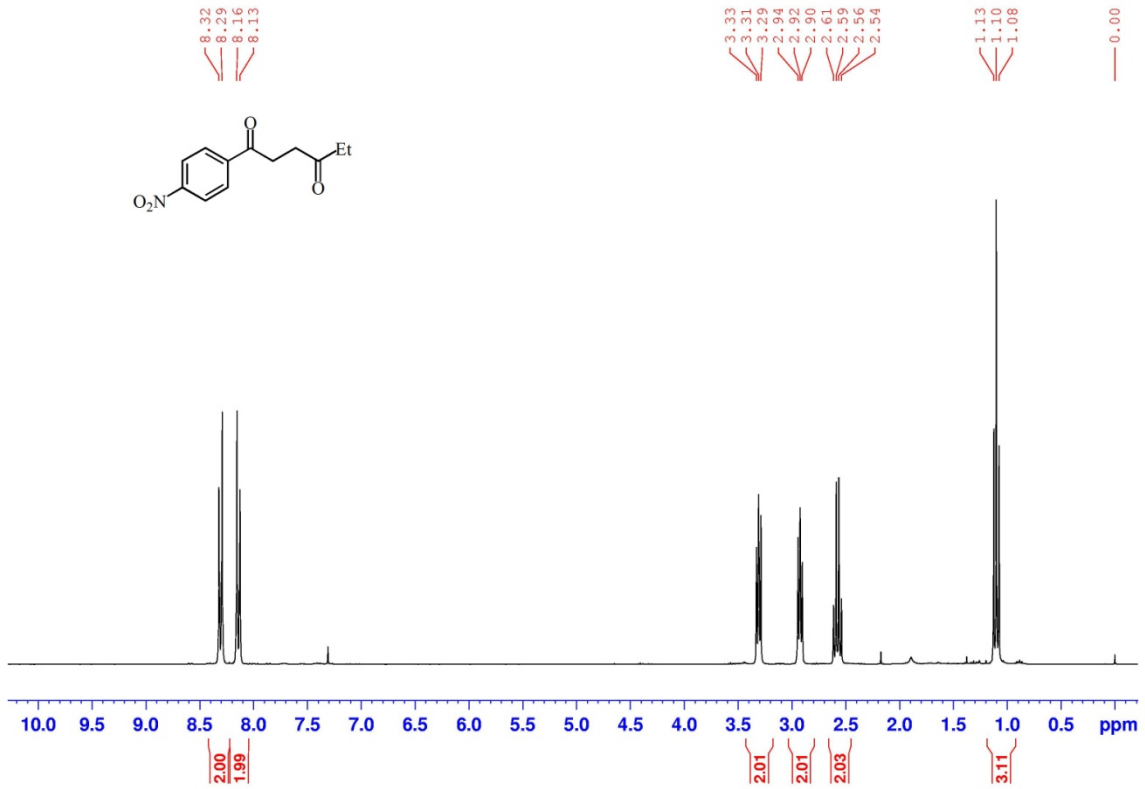
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2.54
1.13
1.10
1.08
0.00
O
Et
O
O2N
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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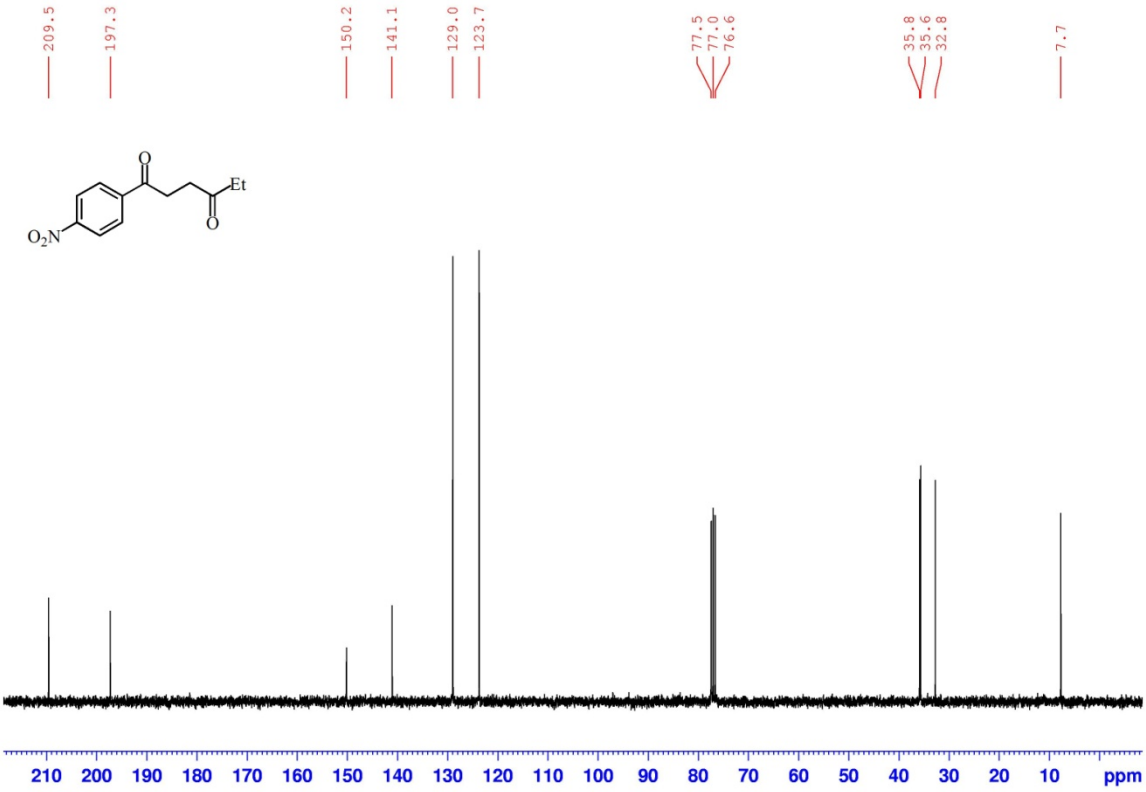
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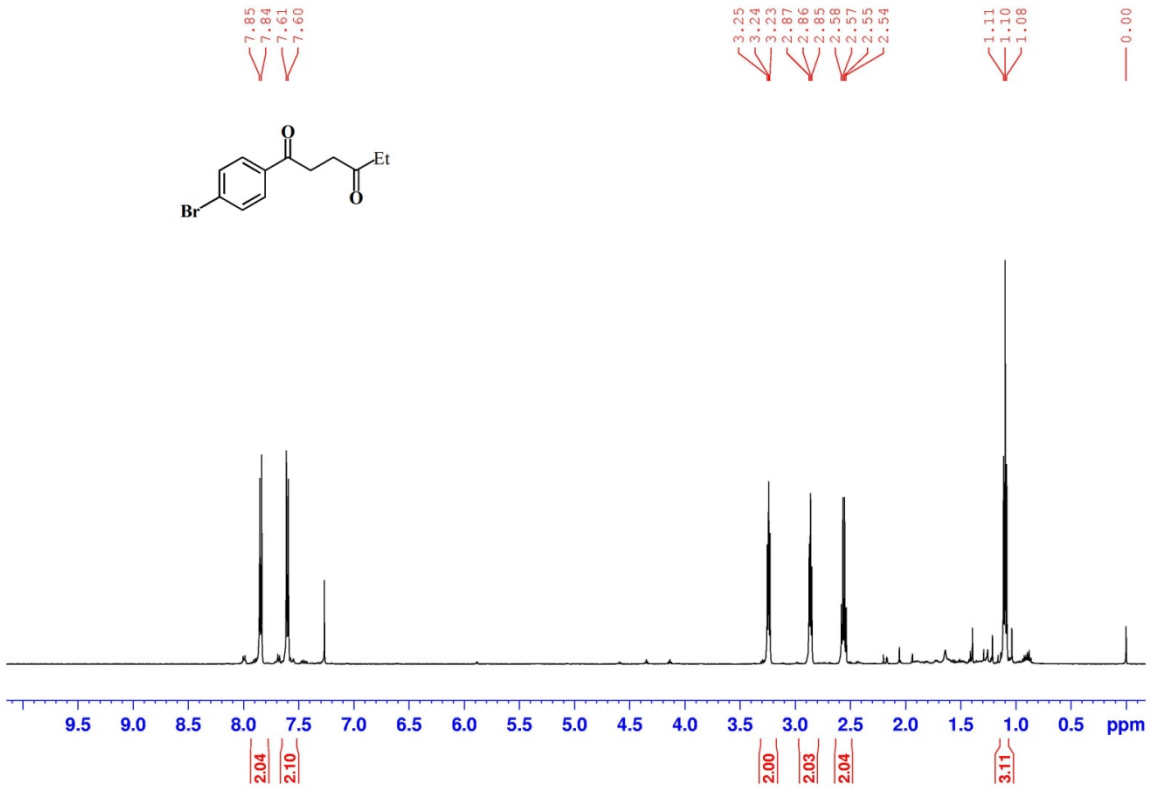
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2.54
1.11
1.10
1.08
0.00
O
Et
O
Br
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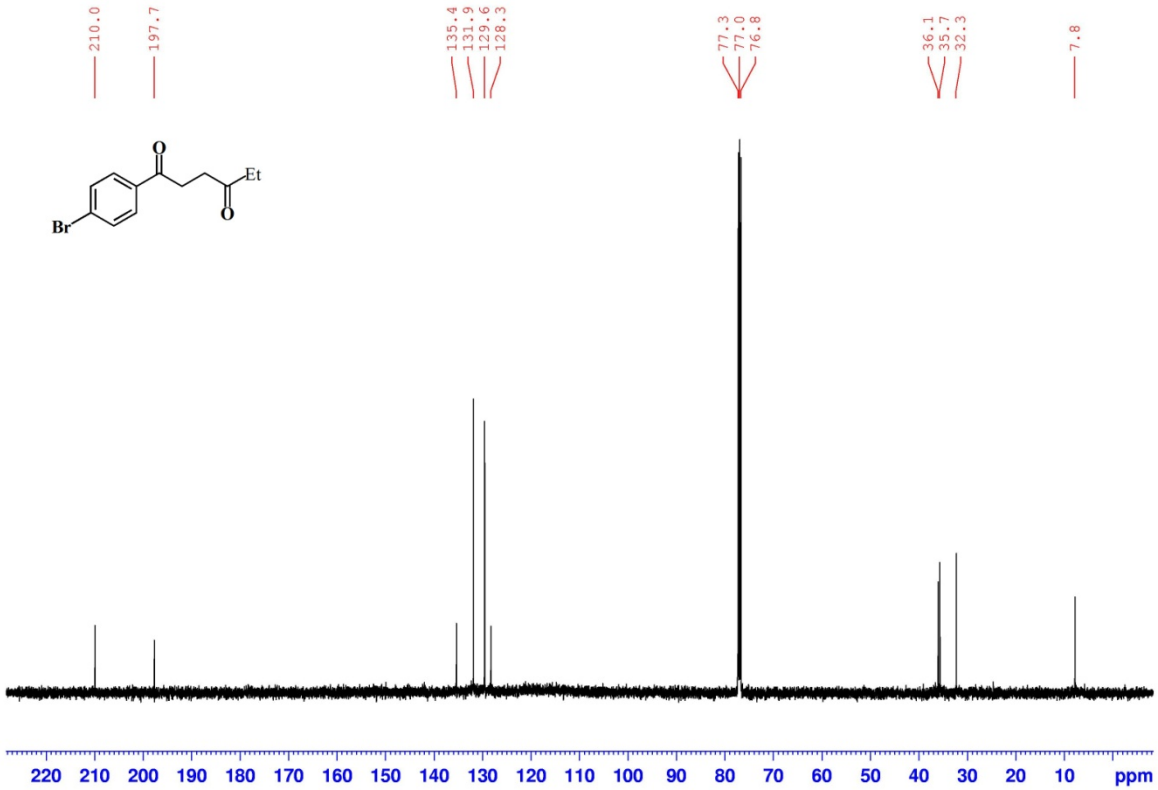
2.00
2.03
2.04
3.11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77.0
76.8
36.1
35.7
32.3
7.8
O
Et
O
Br
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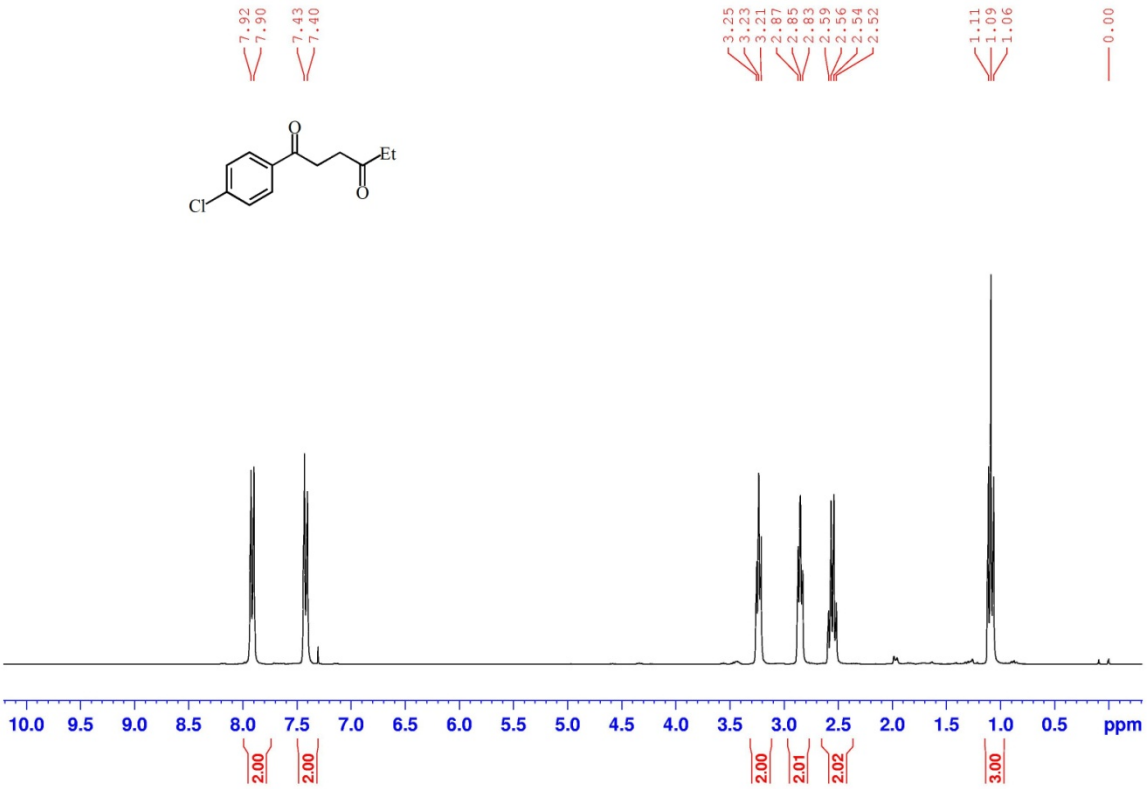
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7.90
7.43
7.40
3.25
3.23
3.21
2.87
2.85
2.83
2.59
2.56
2.54
2.52
1.11
1.09
1.06
0.00
O
Et
O
Cl
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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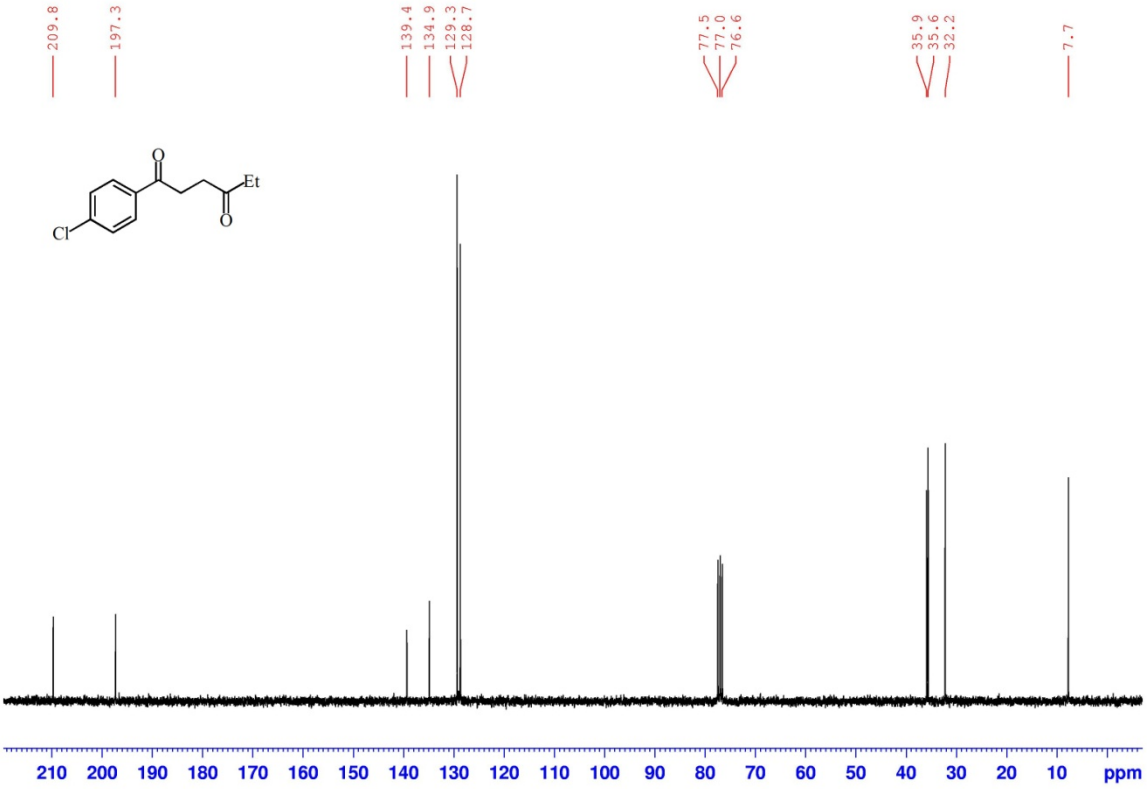
2.00
2.00
2.01
2.02
3.00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197.3
139.4
134.9
129.3
128.7
77.5
77.0
76.6
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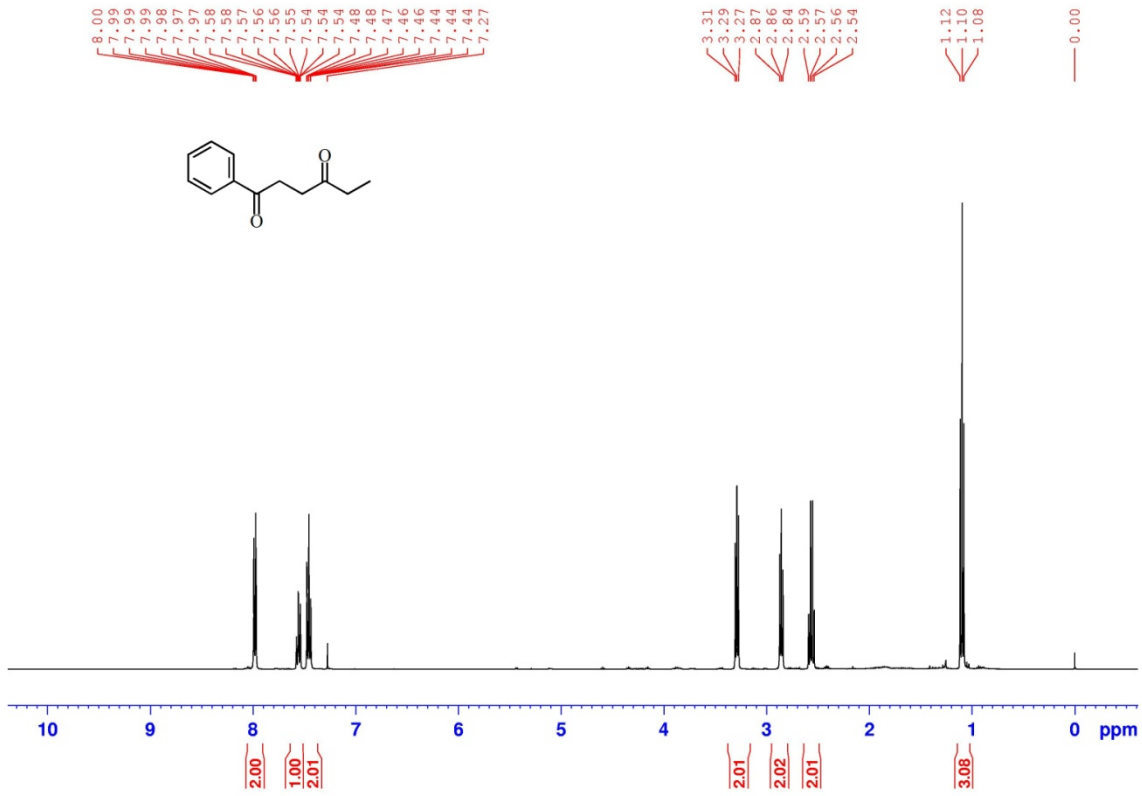
35.6
32.2
7.7
O
Et
O
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.99
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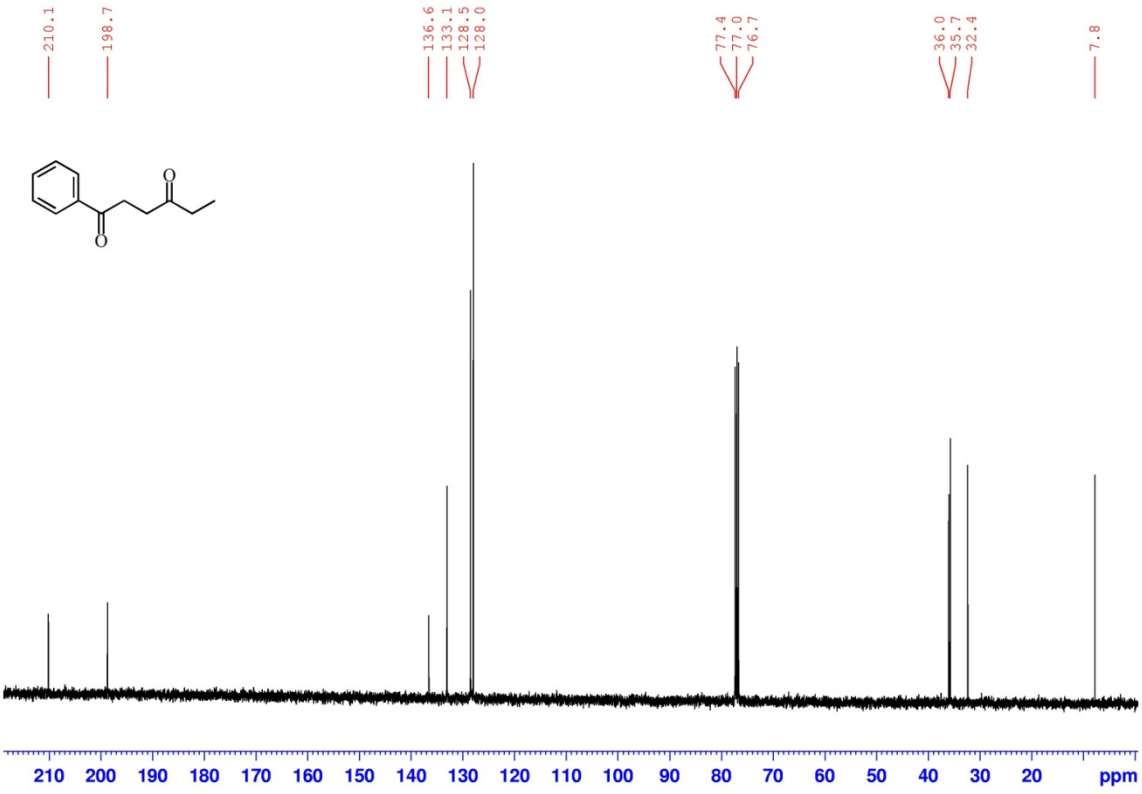
7.58
7.58
7.57
7.56
7.56
7.55
7.54
7.54
7.54
7.48
7.48
7.47
7.46
7.46
7.44
7.44
7.44
7.27
3.31
3.29
3.27
2.87
2.86
2.84
2.59
2.57
2.56
2.54
1.12
1.10
1.08
0.00
O
O
10
9
8
7
6
5
4
3
2
1
0
ppm
2.00
1.00
2.01
2.01
2.02
2.01
3.08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198.7
136.6
133.1
128.5
128.0
77.4
77.0
76.7
36.0
35.7
32.4
7.8
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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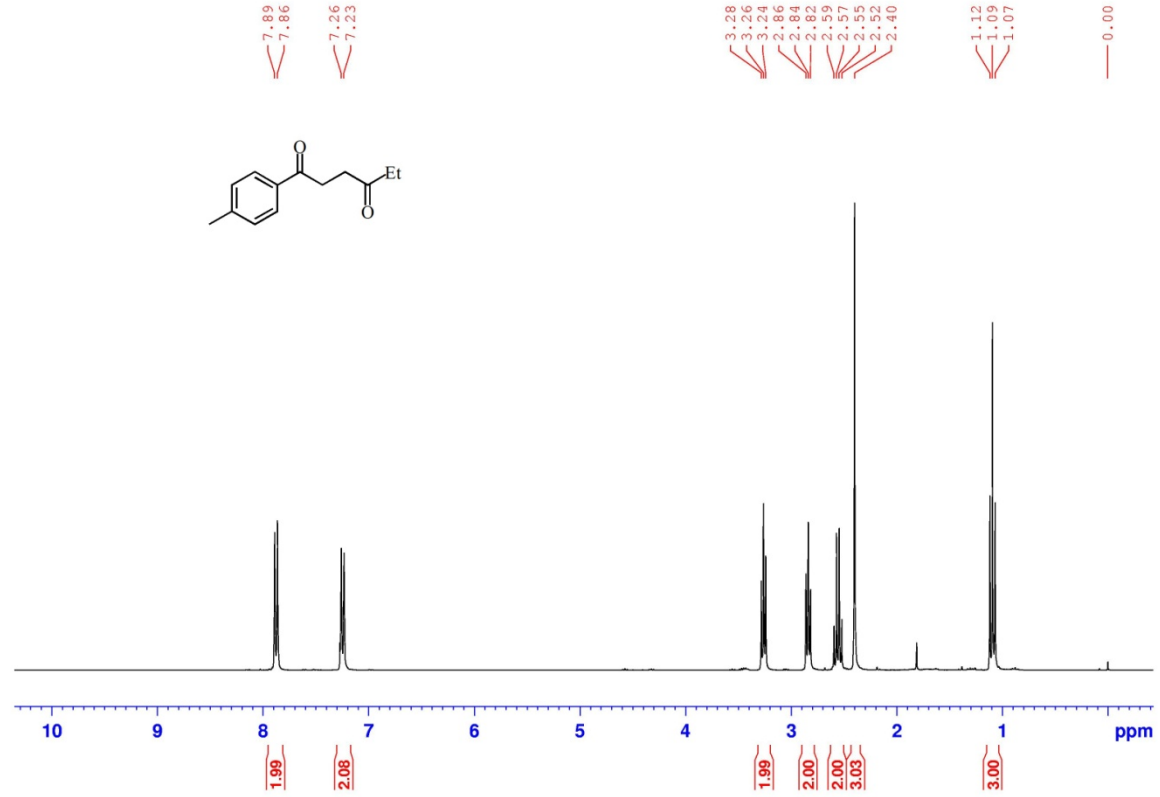
50
40
30
20
ppm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7.86
7.26
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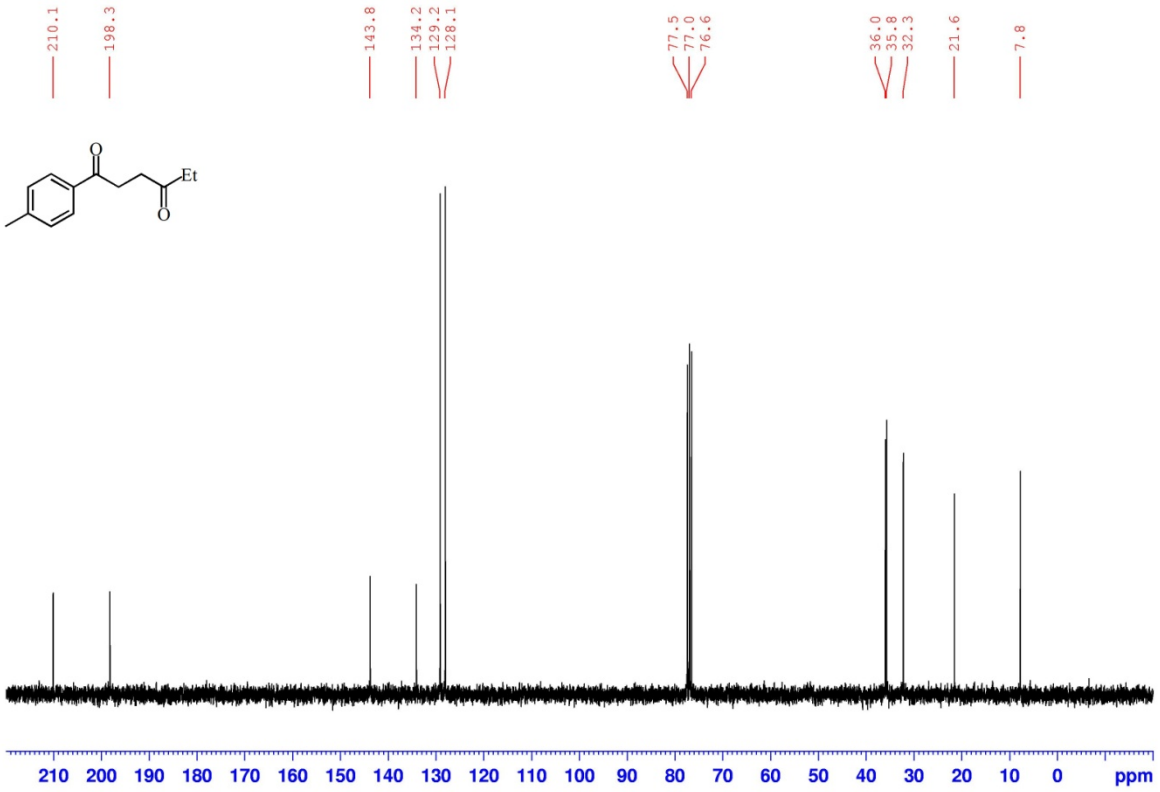
3.26
3.24
2.86
2.84
2.82
2.59
2.57
2.55
2.52
2.40
1.12
1.09
1.07
0.00
O
Et
O
10
9
8
7
6
5
4
3
2
1
ppm
1.99
2.08
1.99
2.00
2.00
3.03
3.00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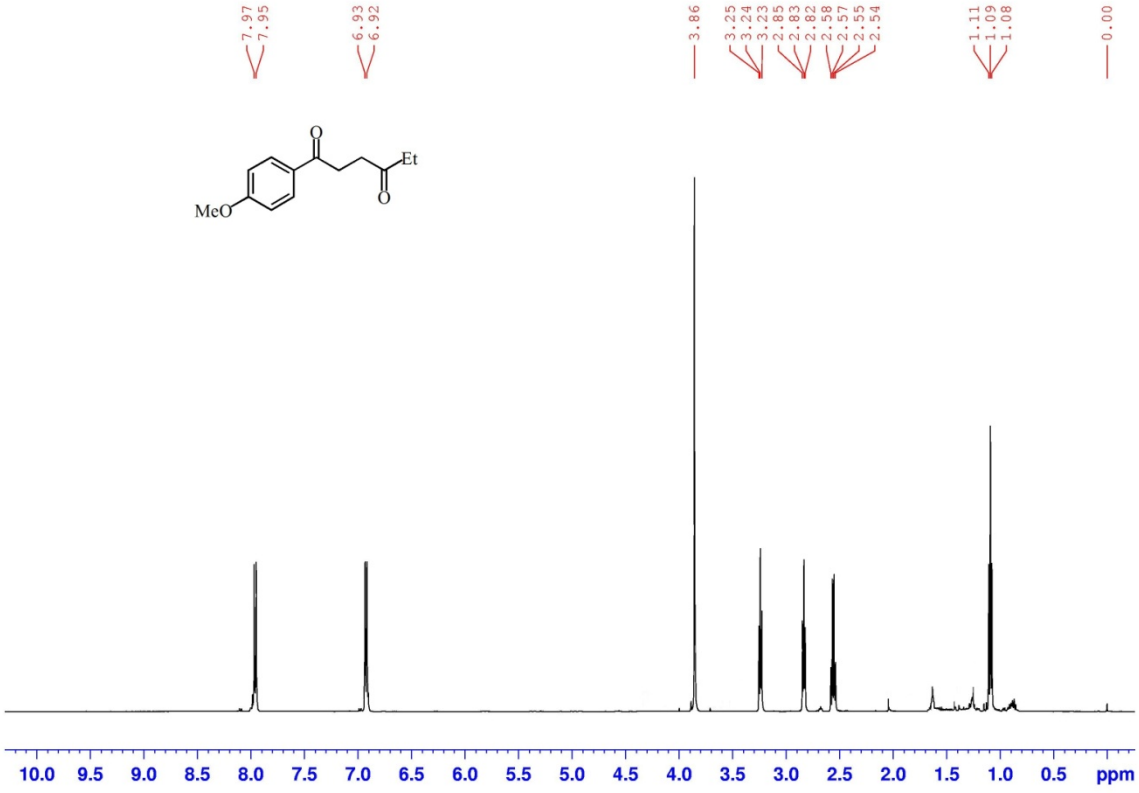
143.8
134.2
129.2
128.1
77.5
77.0
76.6
36.0
35.8
32.3
21.6
7.8
O
Et
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm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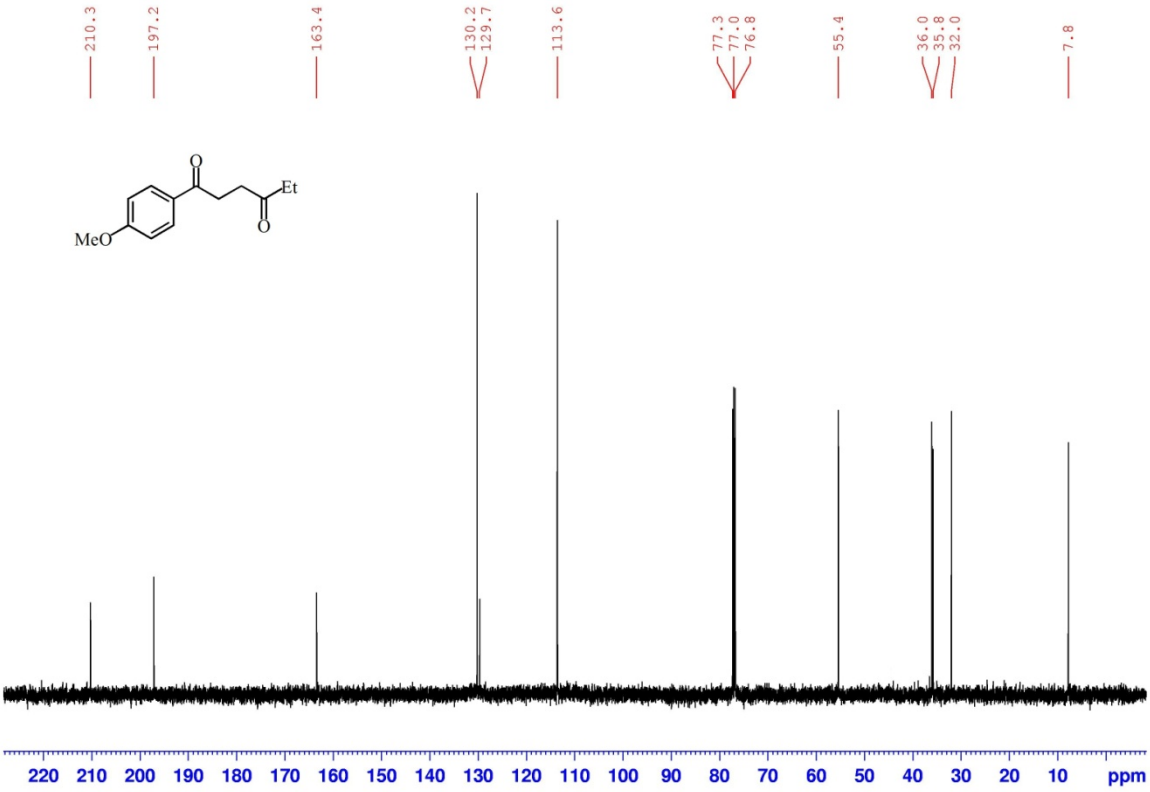
7.95
6.93
6.92
3.86
3.25
3.24
3.23
2.85
2.83
2.82
2.58
2.57
2.55
2.54
1.11
1.09
1.08
0.00
O
Et
O
MeO
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm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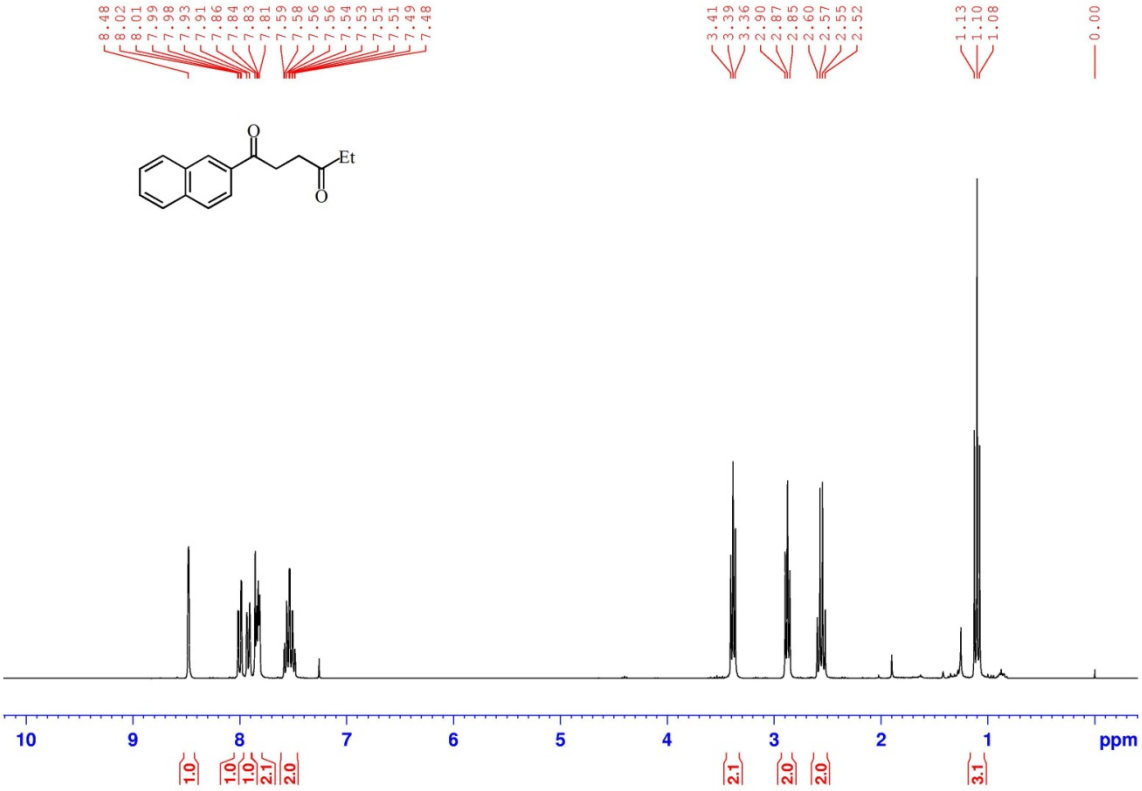
197.2
163.4
130.2
129.7
113.6
77.3
77.0
76.8
55.4
36.0
35.8
32.0
7.8
O
Et
O
MeO
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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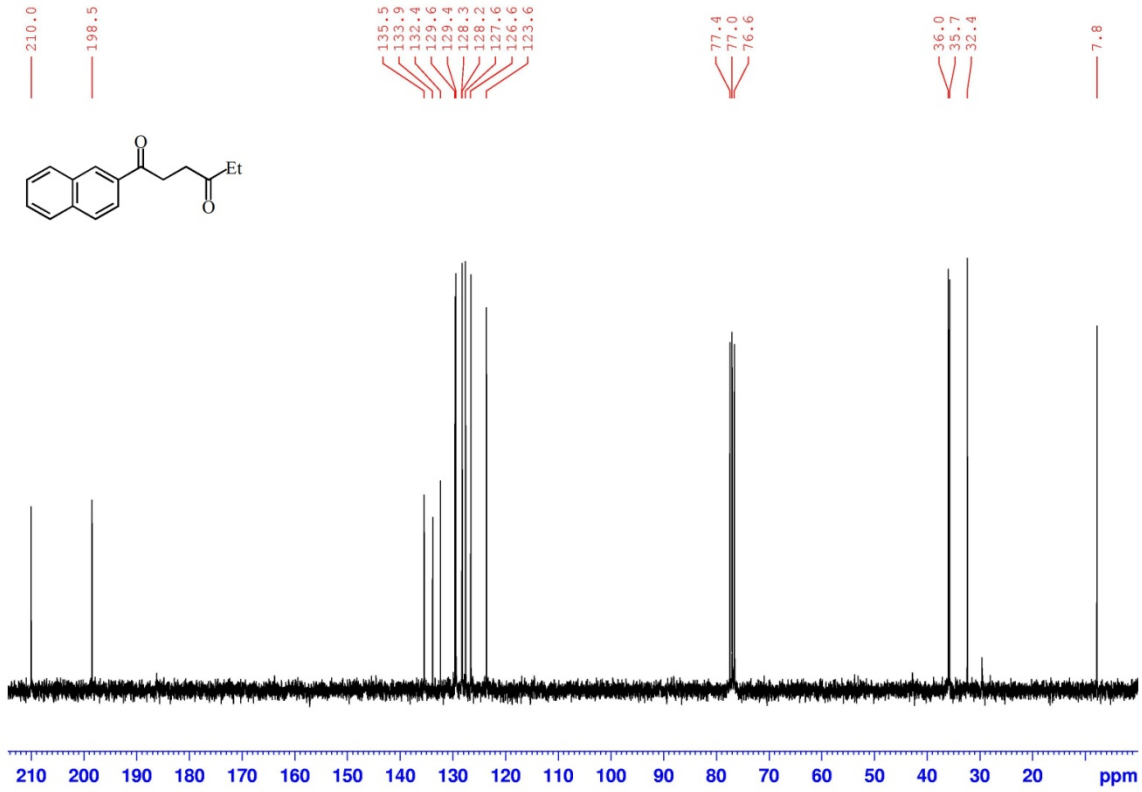
8.02
8.01
7.99
7.98
7.93
7.91
7.86
7.84
7.83
7.81
7.59
7.58
7.56
7.56
7.54
7.53
7.51
7.51
7.49
7.48
3.41
3.39
3.36
2.90
2.87
2.85
2.60
2.57
2.55
2.52
1.13
1.10
1.08
0.00
O
Et
O
10
9
8
7
6
5
4
3
2
1
ppm
1.0
1.0
1.0
2.1
2.0
2.1
2.0
2.0
3.1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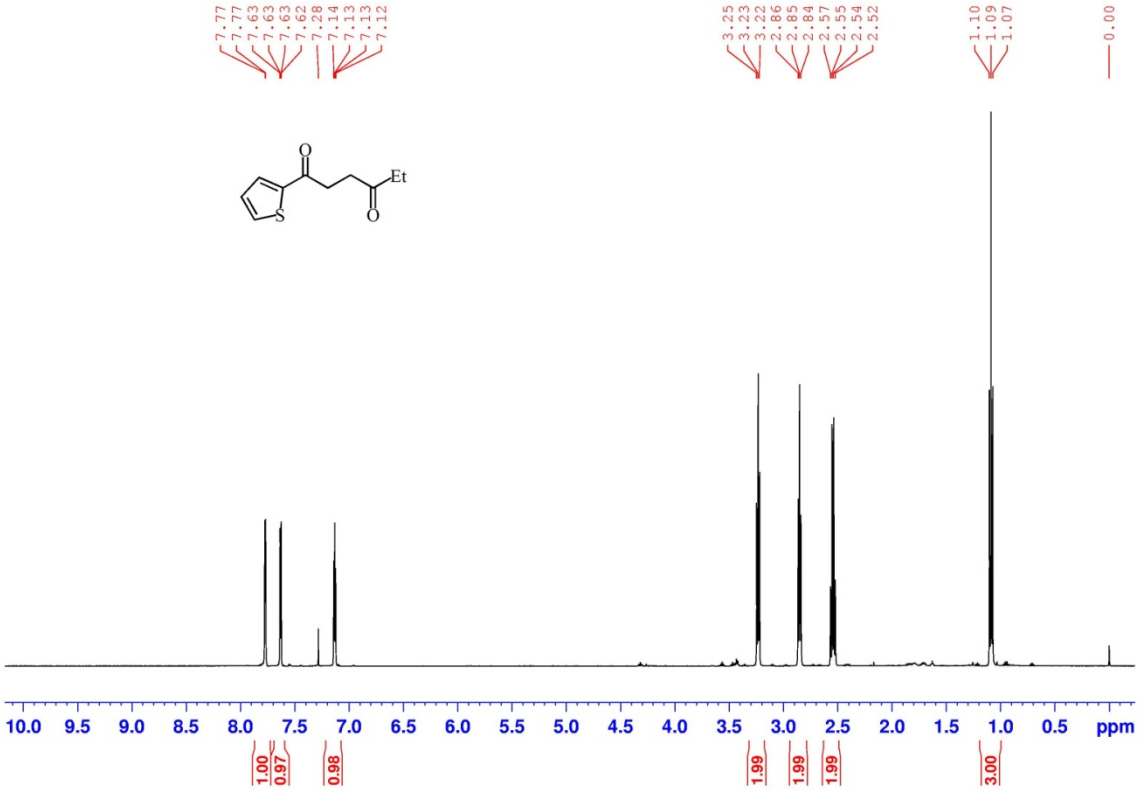
135.5
133.9
132.4
129.6
129.4
128.3
128.2
127.6
126.6
123.6
77.4
77.0
76.6
36.0
35.7
32.4
7.8
O
Et
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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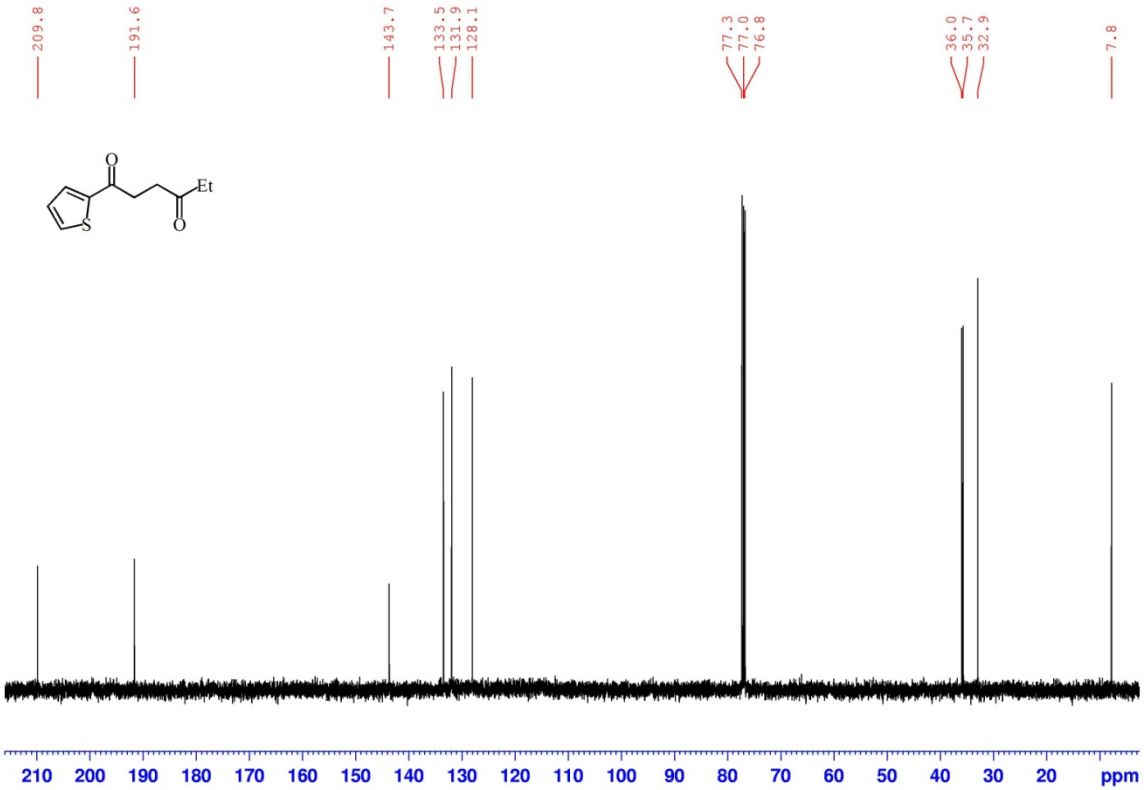
7.77
7.63
7.63
7.63
7.62
7.28
7.14
7.13
7.13
7.12
3.25
3.23
3.22
2.86
2.85
2.84
2.57
2.55
2.54
2.52
1.10
1.09
1.07
0.00
O
Et
S
O
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
0.97
0.98
1.99
1.99
1.99
3.00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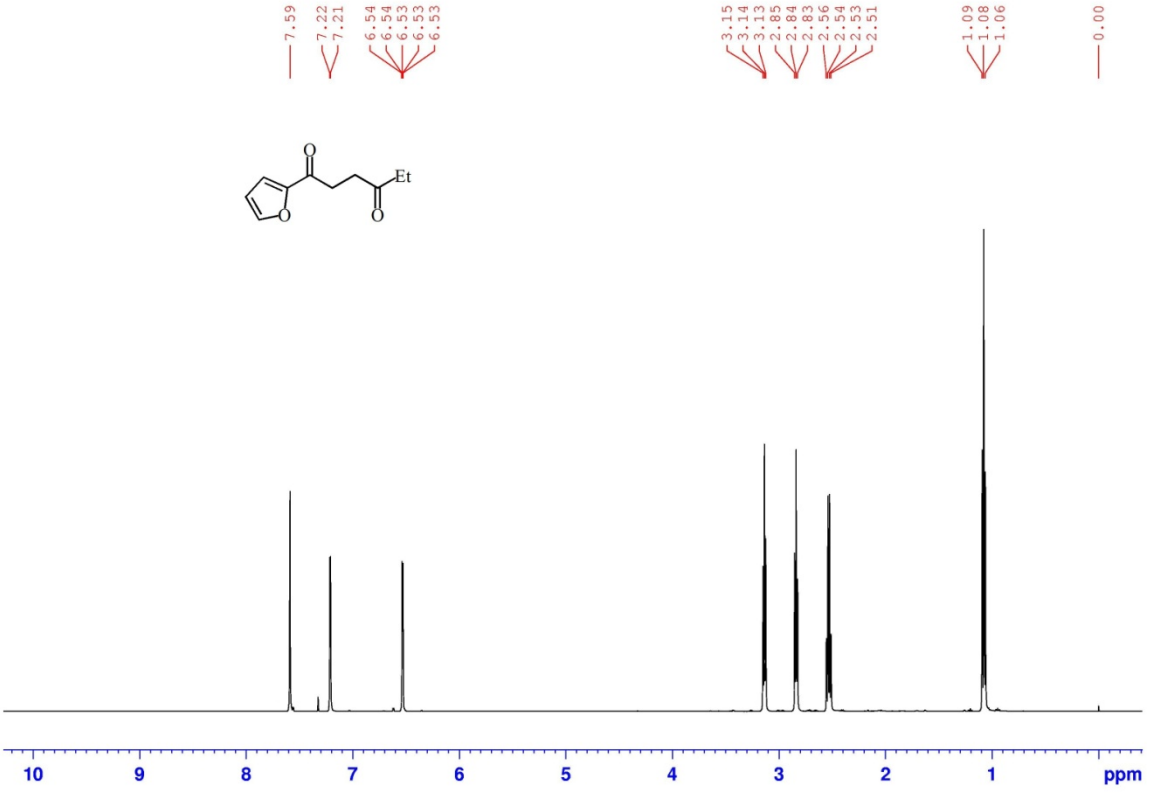
191.6
143.7
133.5
131.9
128.1
77.3
77.0
76.8
36.0
35.7
32.9
7.8
O
Et
S
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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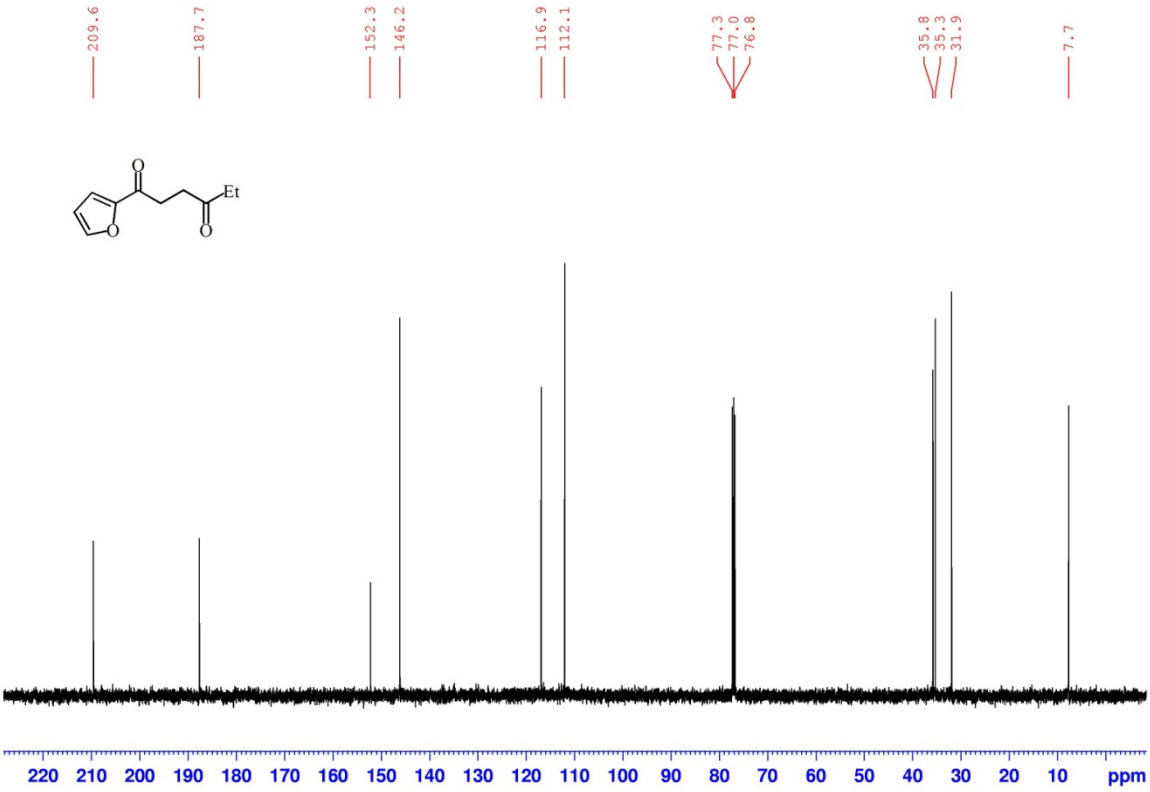
7.22
7.21
6.54
6.54
6.53
6.53
6.53
3.15
3.14
3.13
2.85
2.84
2.83
2.56
2.54
2.53
2.51
1.09
1.08
1.06
0.00
O
O
Et
O
10
9
8
7
6
5
4
3
2
1
ppm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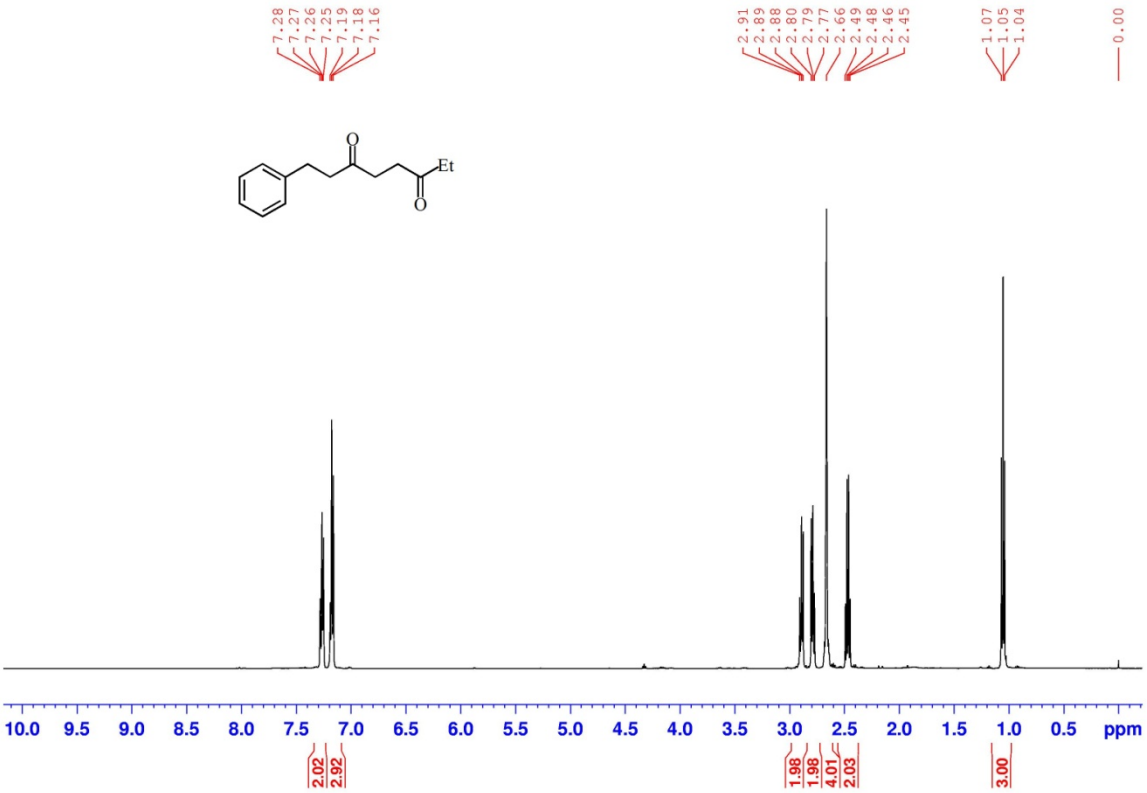
187.7
152.3
146.2
116.9
112.1
77.3
77.0
76.8
35.8
35.3
31.9
7.7
O
O
Et
O
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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7.27
7.26
7.25
7.19
7.18
7.16
2.91
2.89
2.88
2.80
2.79
2.77
2.66
2.49
2.48
2.46
2.45
1.07
1.05
1.04
0.00
Et
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
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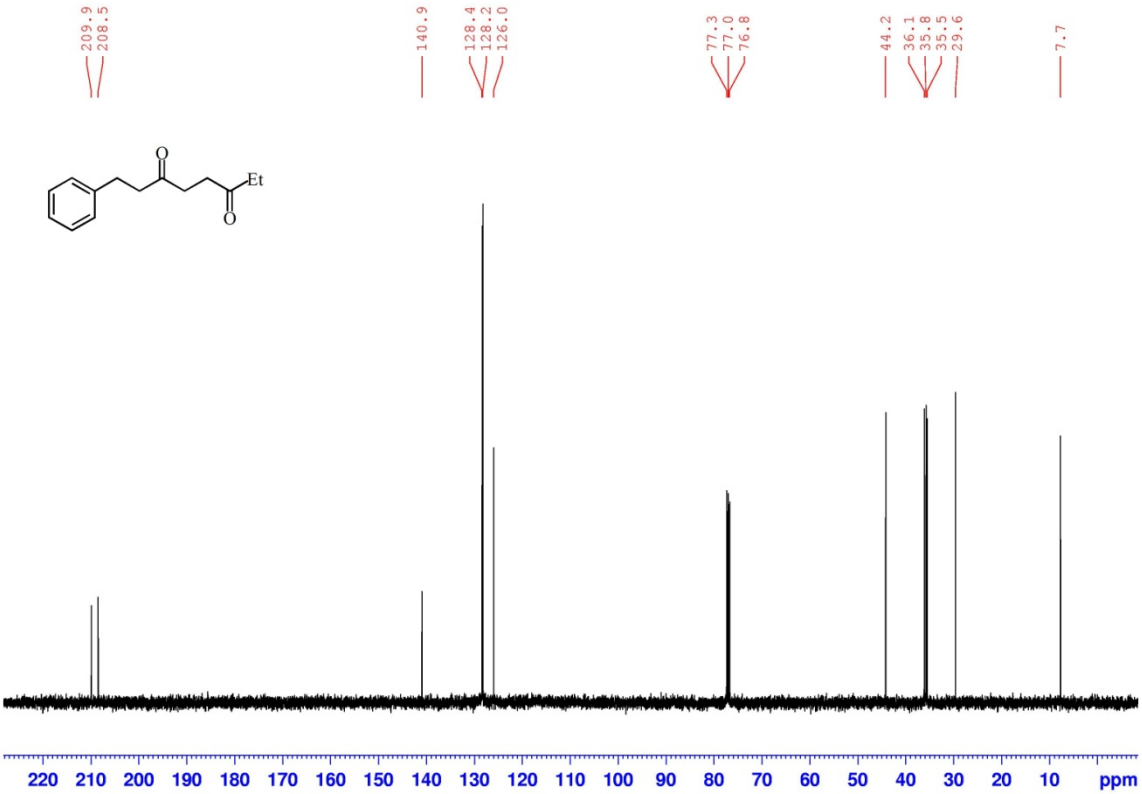
2.92
1.98
1.98
4.01
2.03
3.00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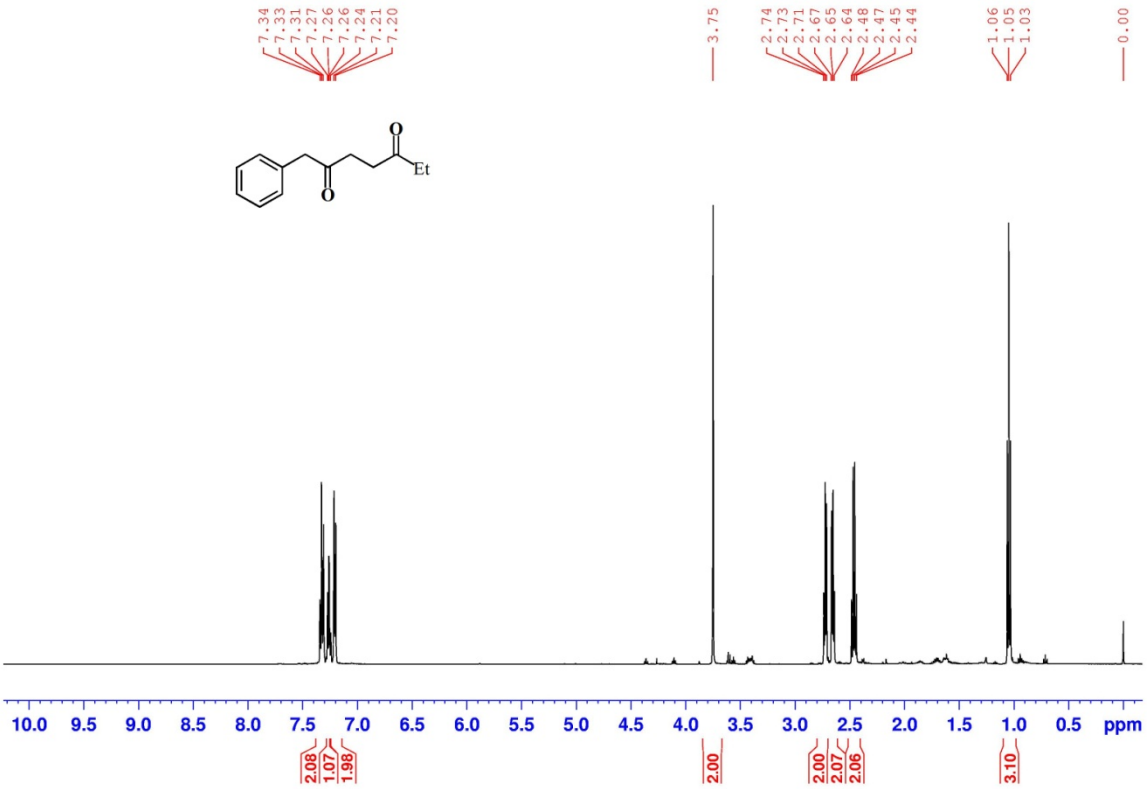
208.5
140.9
128.4
128.2
126.0
77.3
77.0
76.8
44.2
36.1
35.8
35.5
29.6
7.7
Et
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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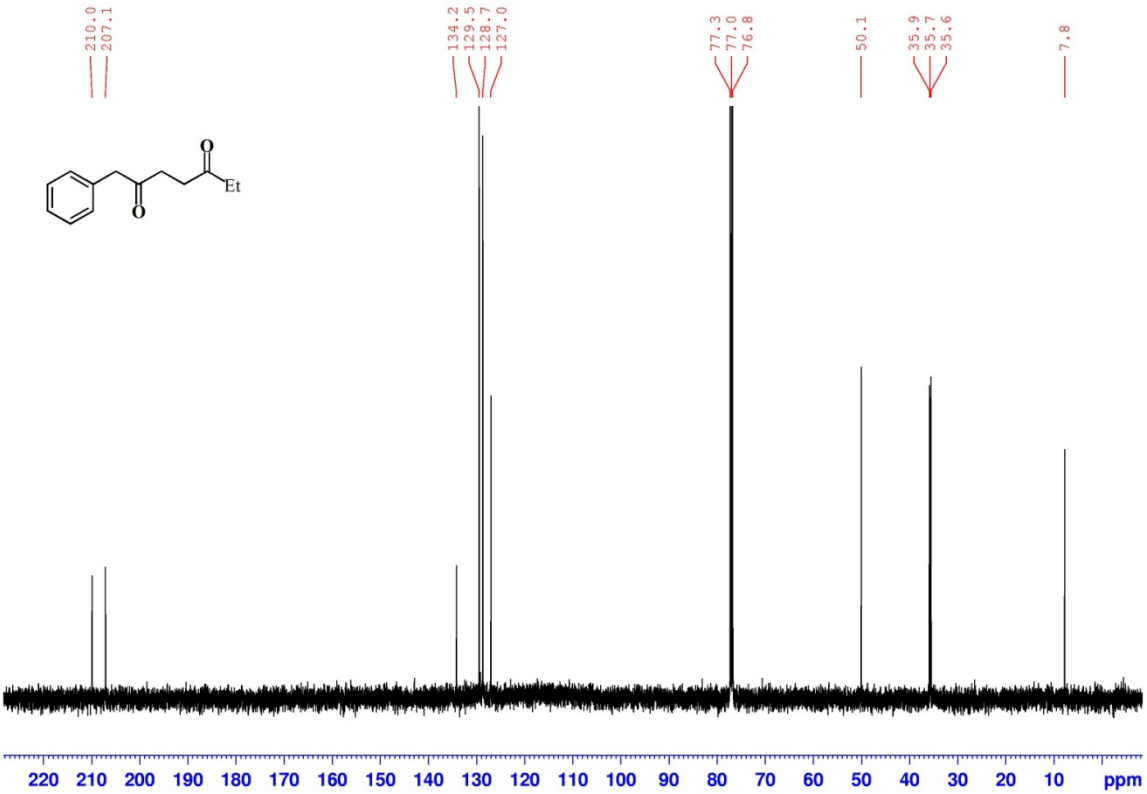
7.33
7.31
7.27
7.26
7.26
7.24
7.21
7.20
3.75
2.74
2.73
2.71
2.67
2.65
2.64
2.48
2.47
2.45
2.44
1.06
1.05
1.03
0.00
O
O
Et
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.08
1.07
1.98
2.00
2.00
2.07
2.06
3.10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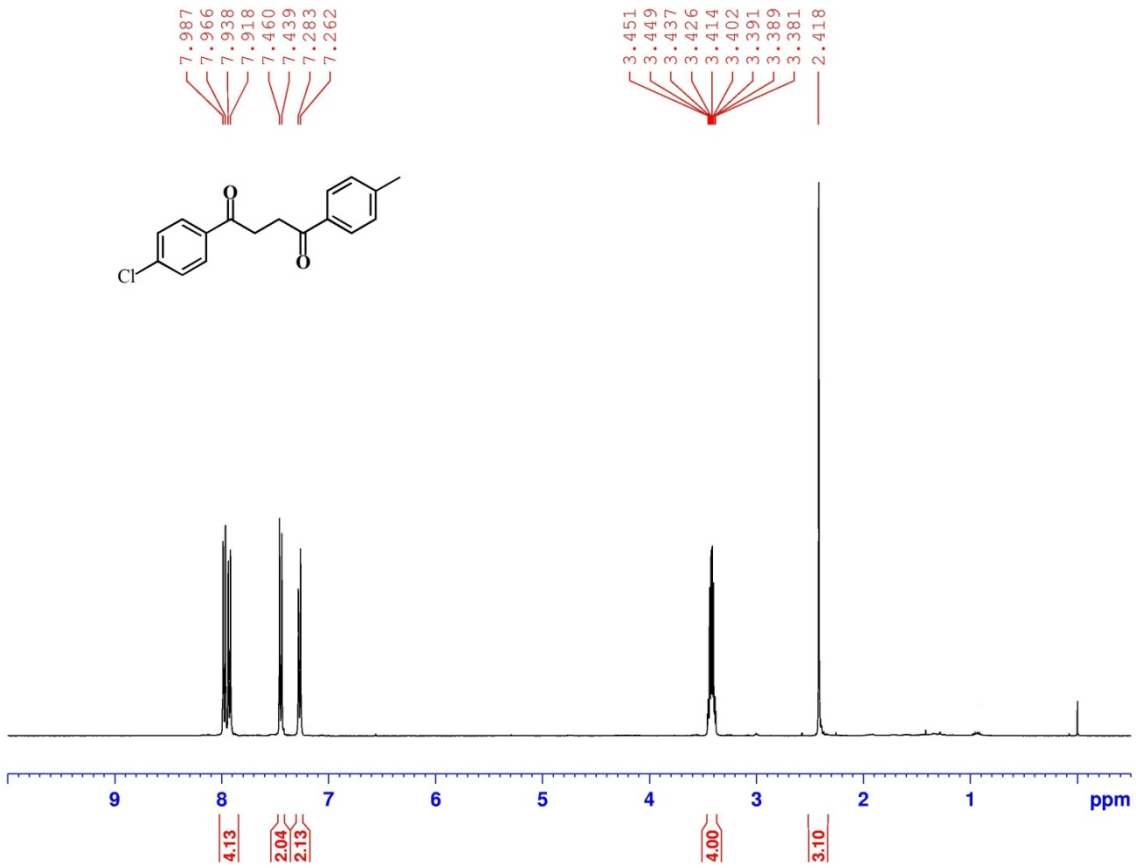
134.2
129.5
128.7
127.0
77.3
77.0
76.8
50.1
35.9
35.7
35.6
7.8
O
O
Et
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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7.918
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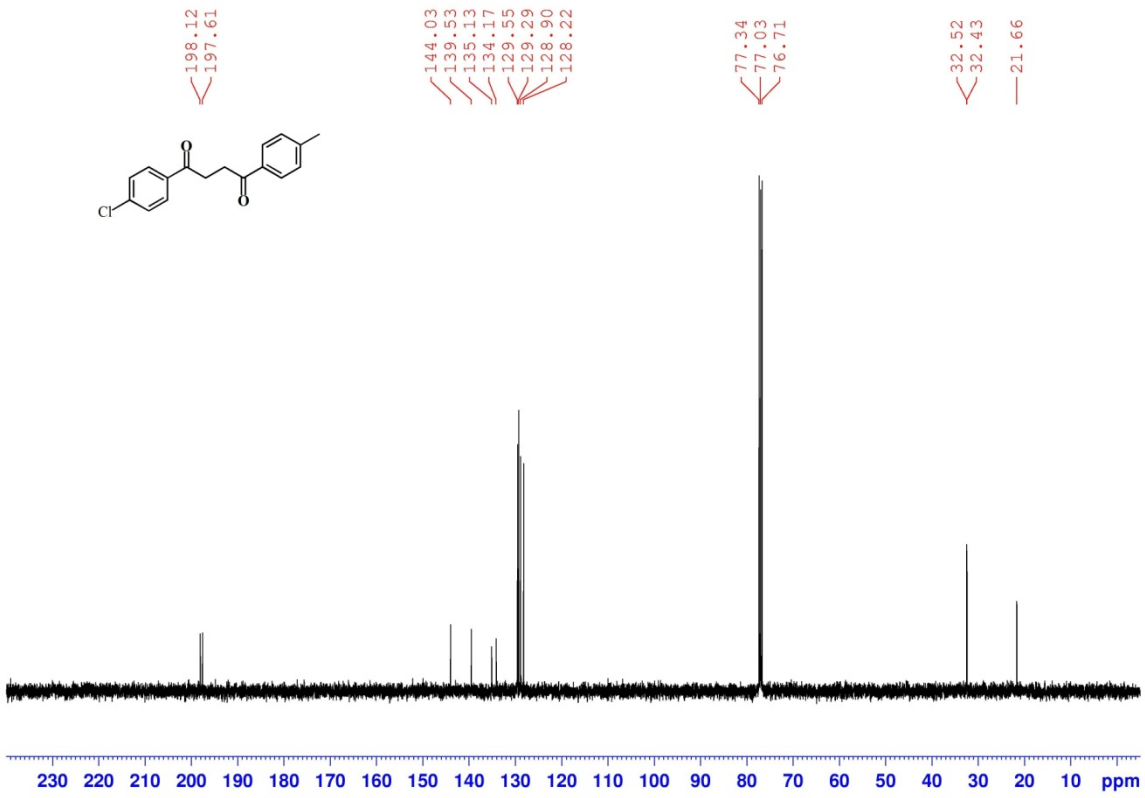
7.439
7.283
7.262
3.451
3.449
3.437
3.426
3.414
3.402
3.391
3.389
3.381
2.418
O
O
Cl
9
8
7
6
5
4
3
2
1
ppm
4.13
2.04
2.13
4.00
3.10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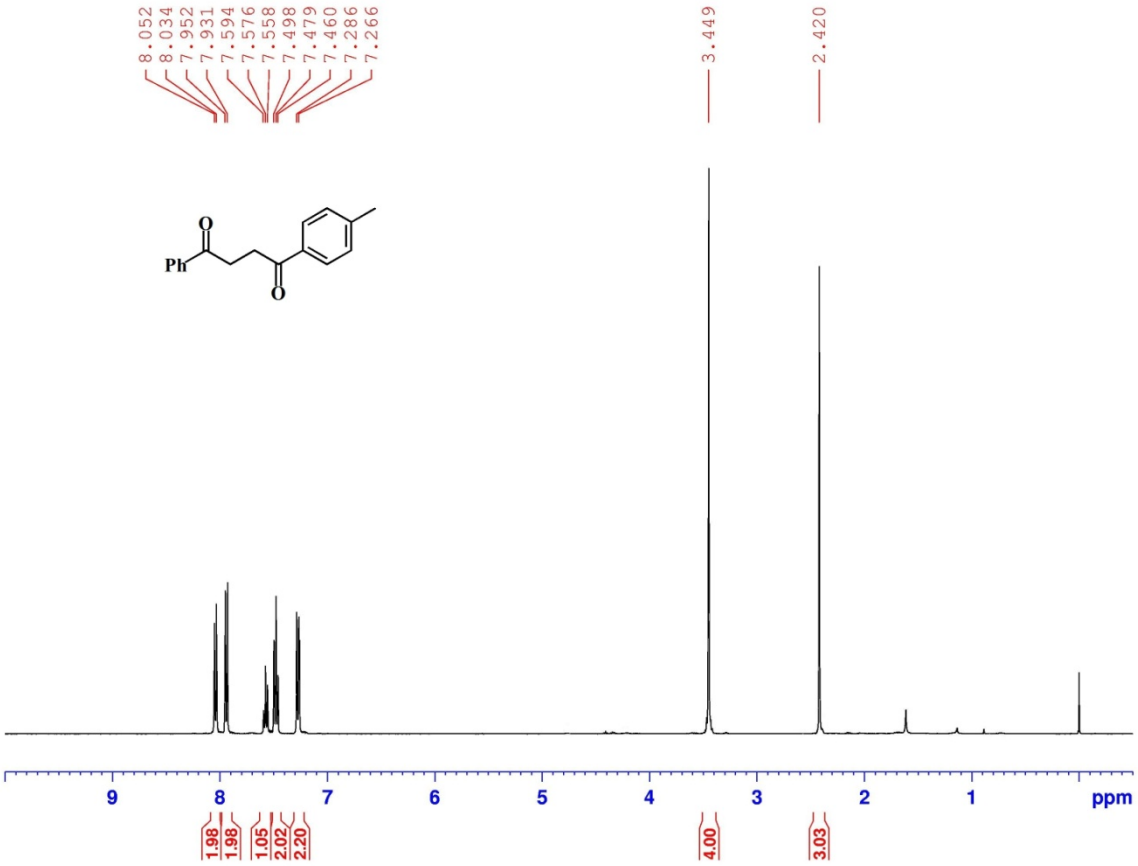
197.61
144.03
139.53
135.13
134.17
129.55
129.29
128.90
128.22
77.34
77.03
76.71
32.52
32.43
21.66
O
O
Cl
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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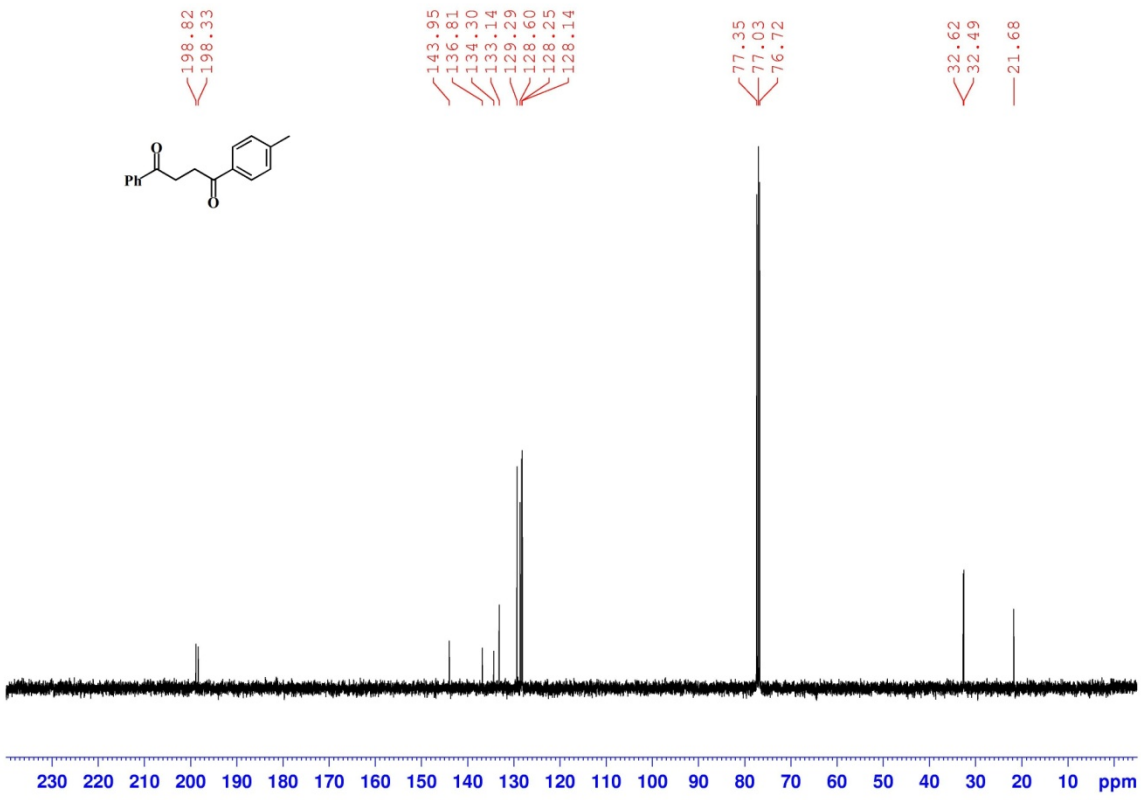
8.034
7.952
7.931
7.594
7.576
7.558
7.498
7.479
7.460
7.286
7.266
3.449
2.420
Ph
O
O
9
8
7
6
5
4
3
2
1
ppm
1.98
1.98
1.05
2.02
2.20
4.00
3.03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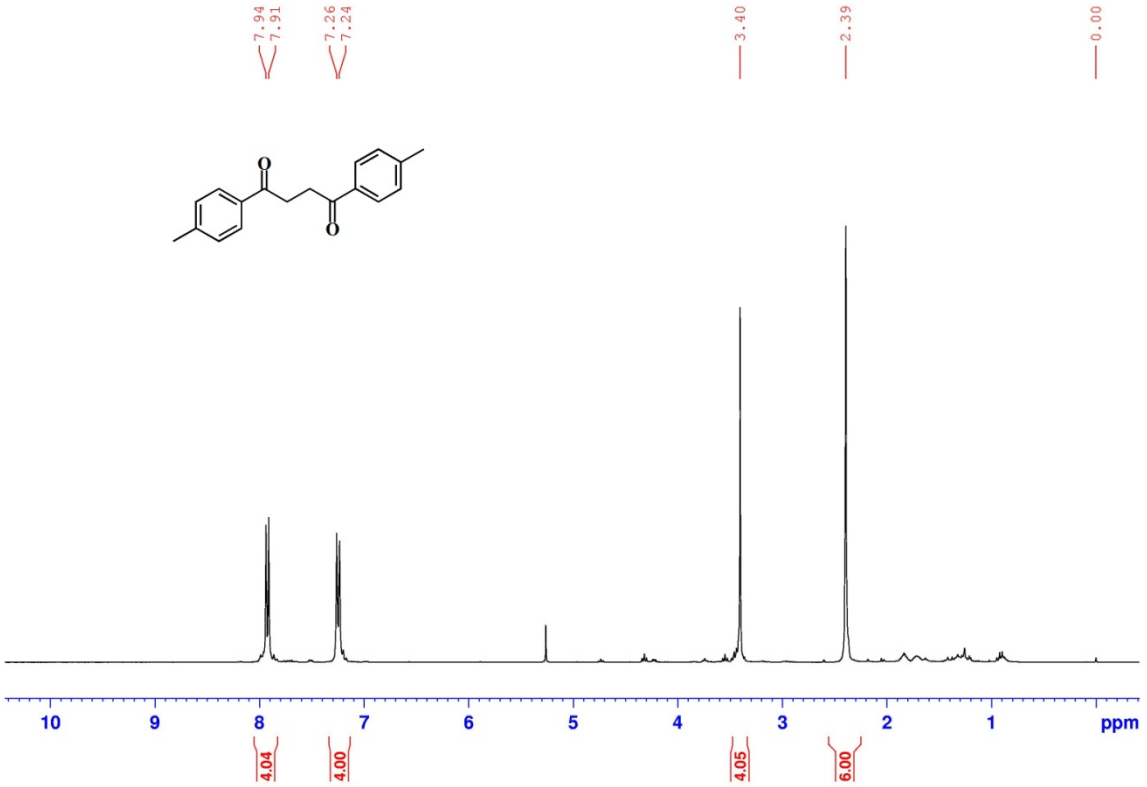
198.33
143.95
136.81
134.30
133.14
129.29
128.60
128.25
128.14
77.35
77.03
76.72
32.62
32.49
21.68
Ph
O
O
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm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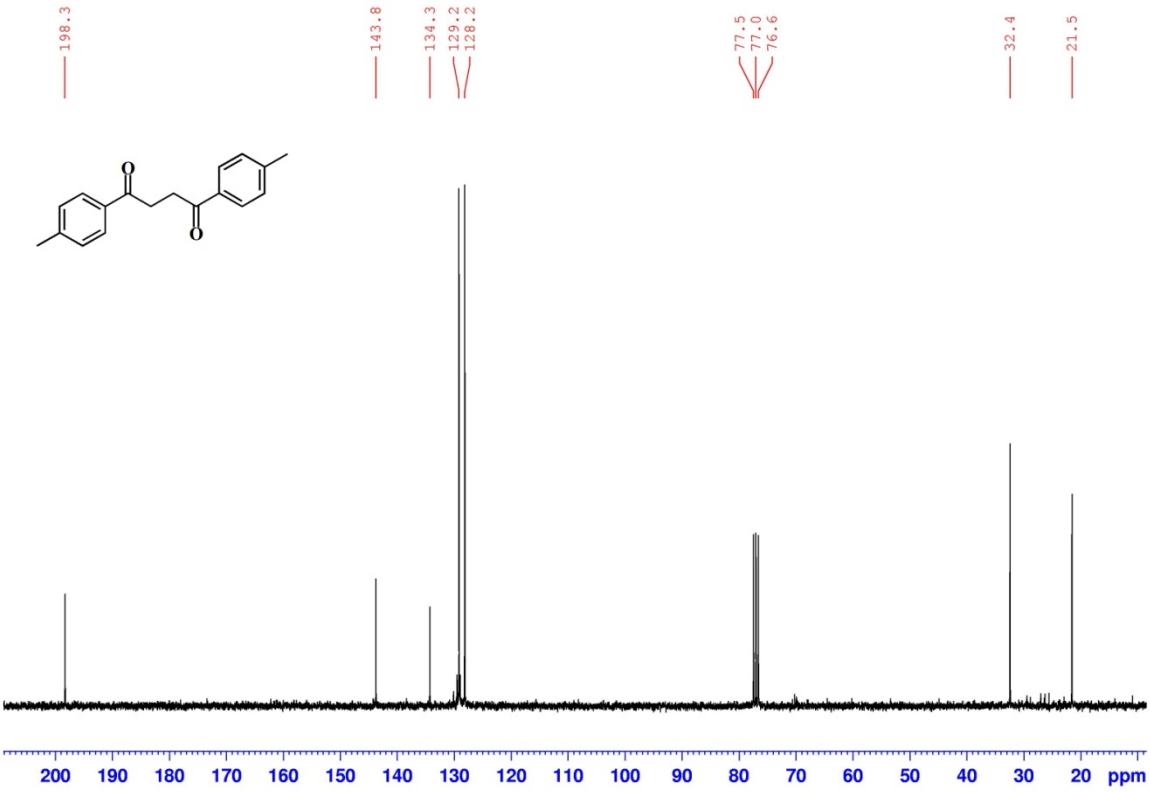
7.91
7.26
7.24
3.40
2.39
0.00
10
9
8
7
6
5
4
3
2
1
ppm
4.04
4.00
4.05
6.00


198.3
143.8
134.3
129.2
128.2
77.5
77.0
76.6
32.4
21.5
200
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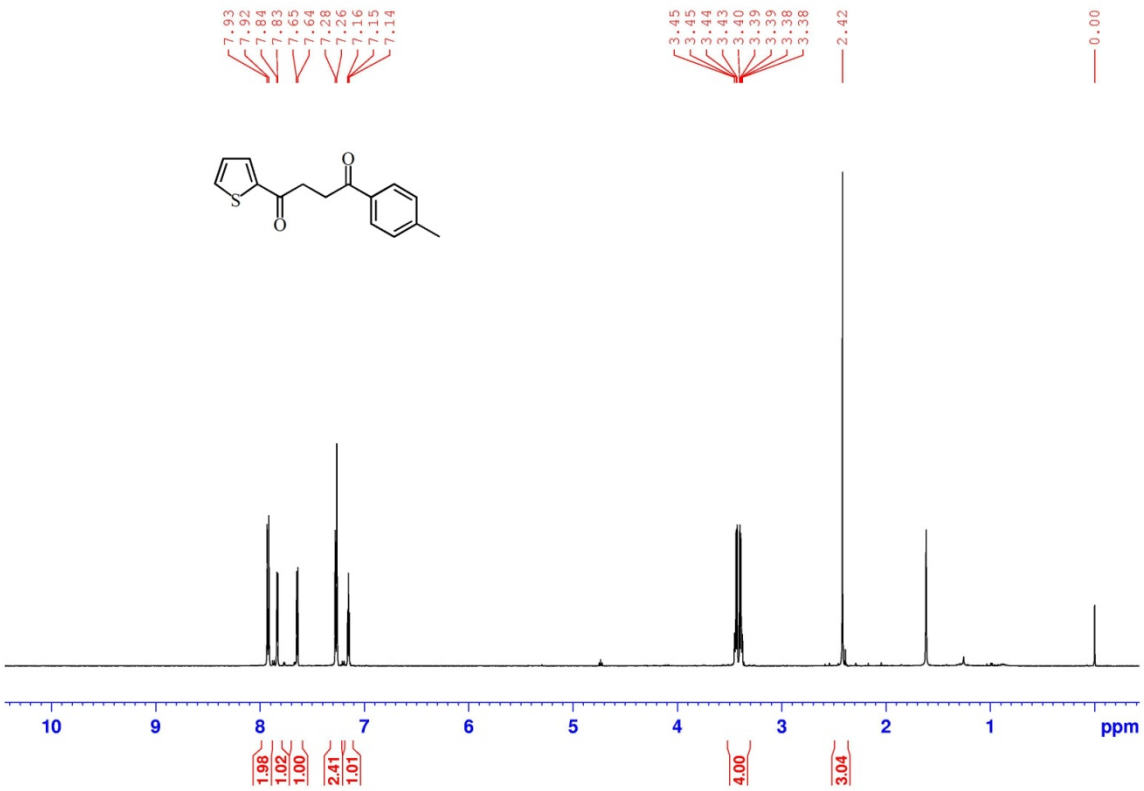
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm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7.92
7.84
7.83
7.65
7.64
7.28
7.26
7.16
7.15
7.14
3.45
3.45
3.44
3.43
3.40
3.39
3.39
3.38
3.38
2.42
0.00
O
S
O
10
9
8
7
6
5
4
3
2
1
ppm
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1.00
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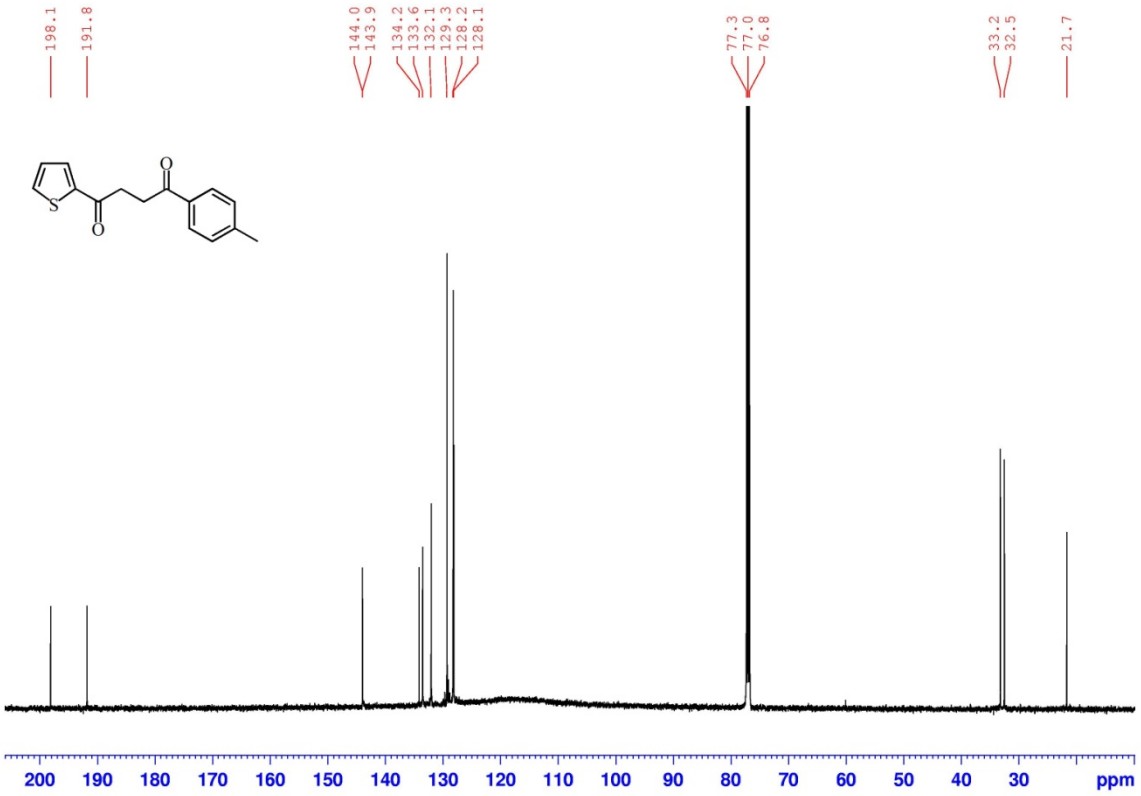
1.01
4.00
3.04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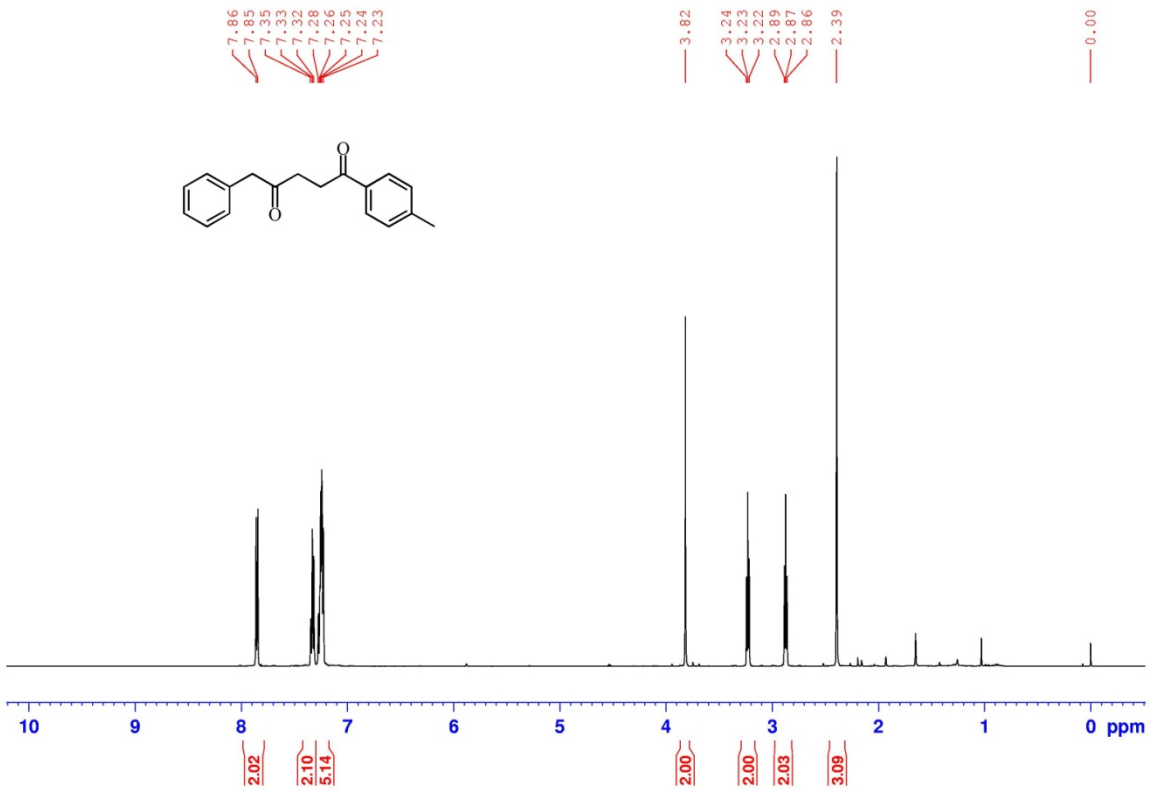
191.8
144.0
143.9
134.2
133.6
132.1
129.3
128.2
128.1
77.3
77.0
76.8
33.2
32.5
21.7
O
S
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm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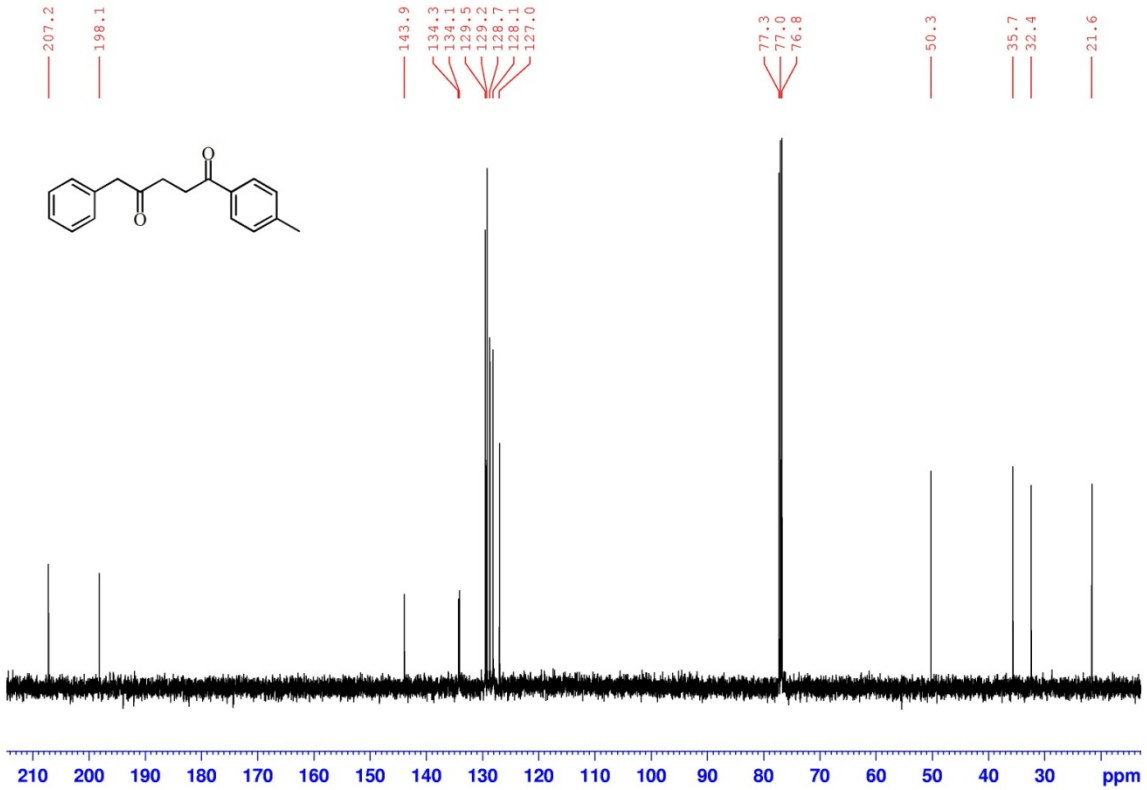
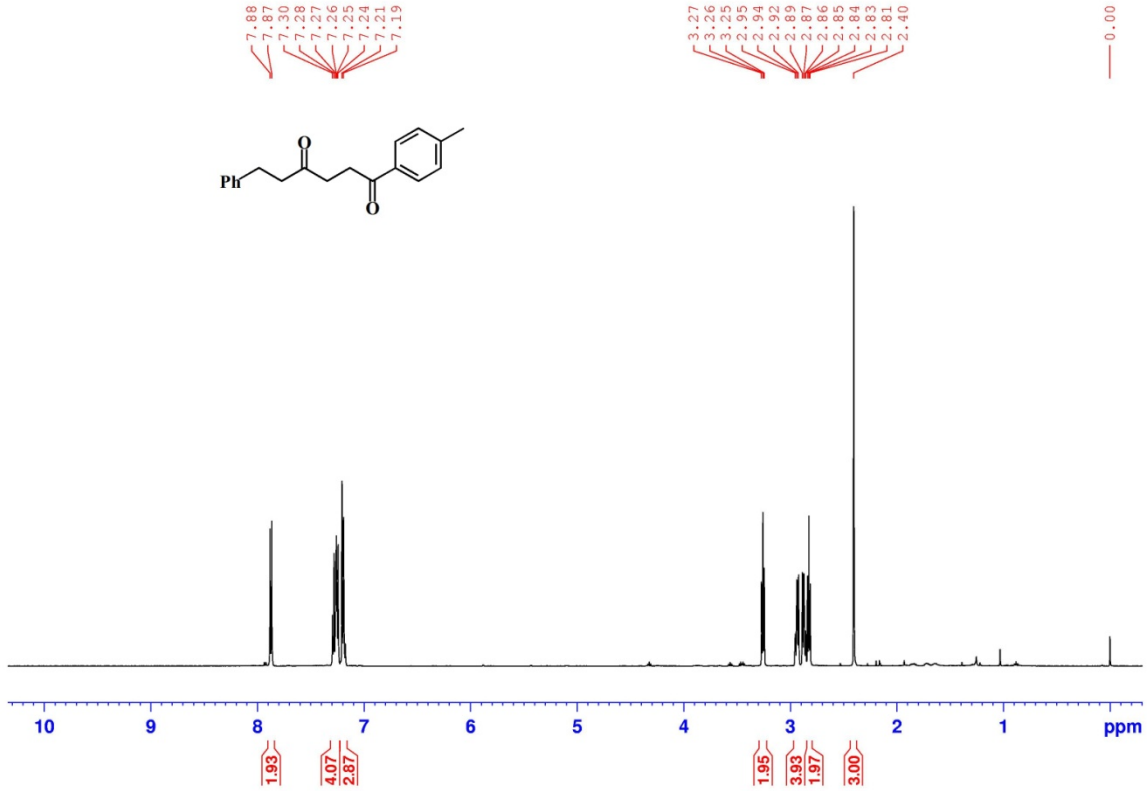
7.85
7.35
7.33
7.32
7.28
7.26
7.25
7.24
7.23
3.82
3.24
3.23
3.22
2.89
2.87
2.86
2.39
0.00
10 9 8 7 6 5 4 3 2 1 0 ppm
2.02
2.10
5.14
2.00
2.00
2.03
3.09


207.2
198.1
143.9
134.3
134.1
129.5
129.2
128.7
128.1
127.0
77.3
77.0
76.8
50.3
35.7
32.4
21.6
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm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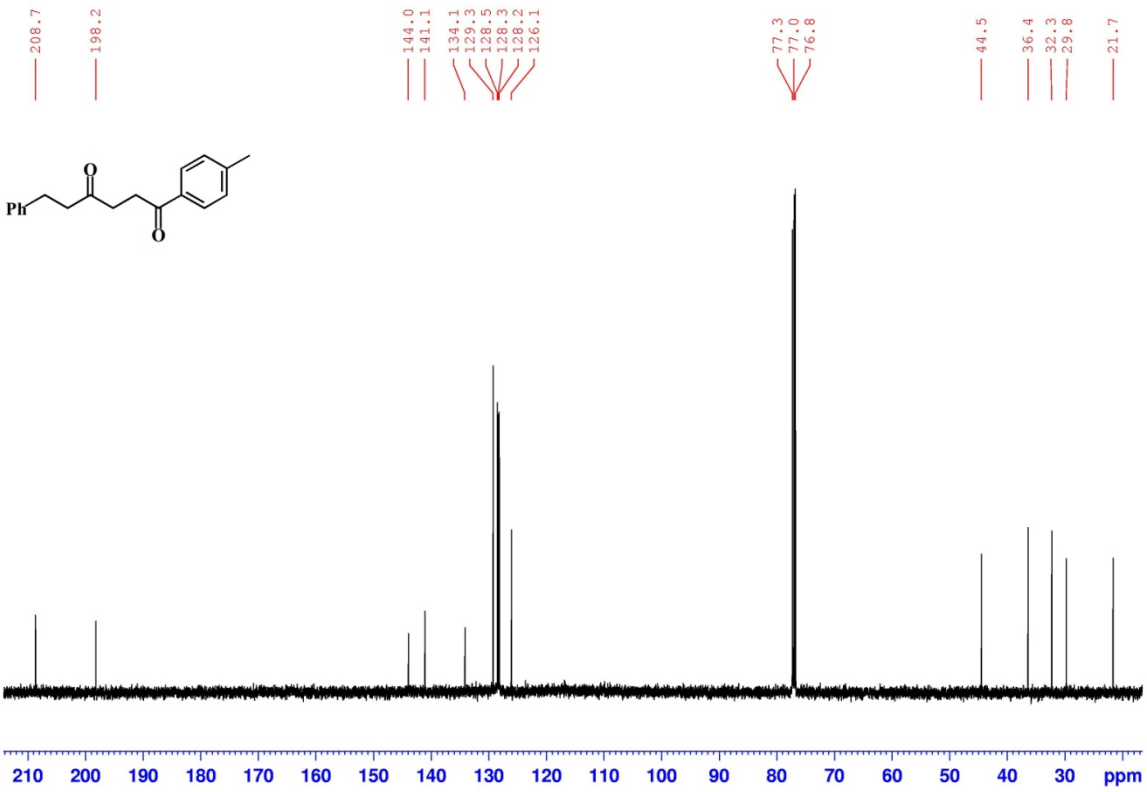
7.87
7.30
7.28
7.27
7.26
7.25
7.24
7.21
7.19
3.27
3.26
3.25
2.95
2.94
2.92
2.89
2.87
2.86
2.85
2.84
2.83
2.81
2.40
0.00
Ph
O
O
10
9
8
7
6
5
4
3
2
1
ppm
1.93
4.07
2.87
1.95
3.93
1.97
3.00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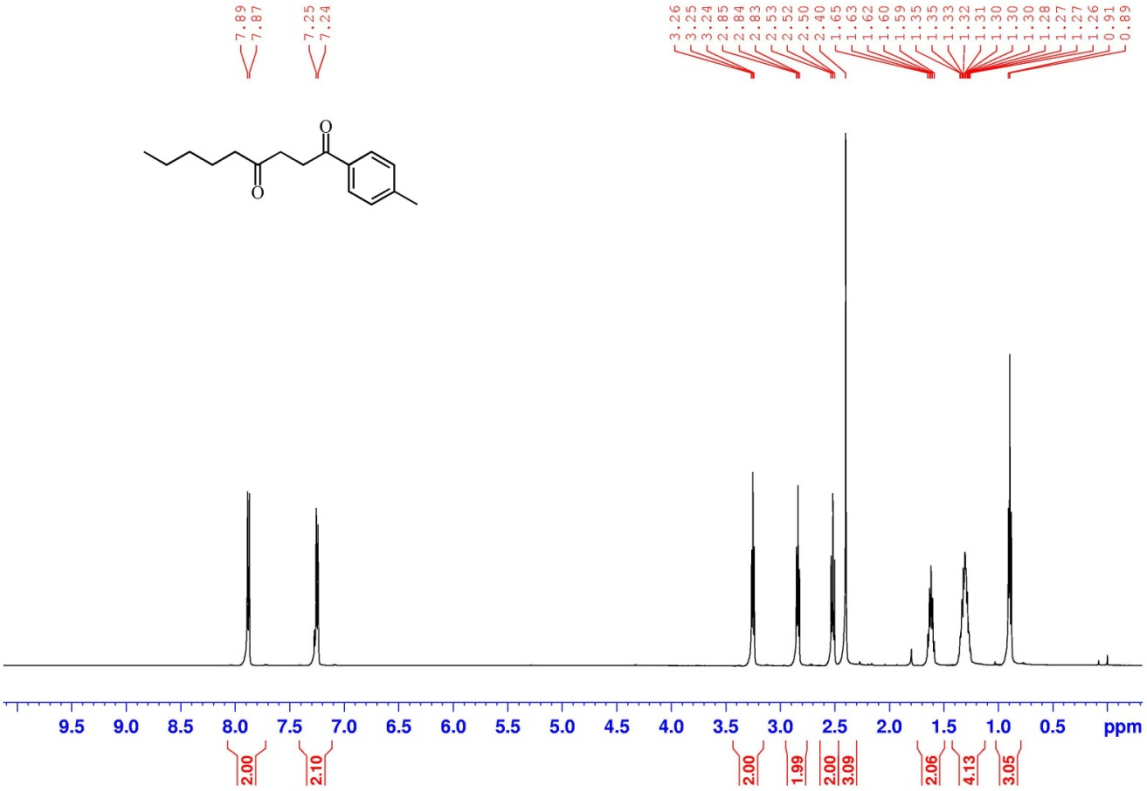
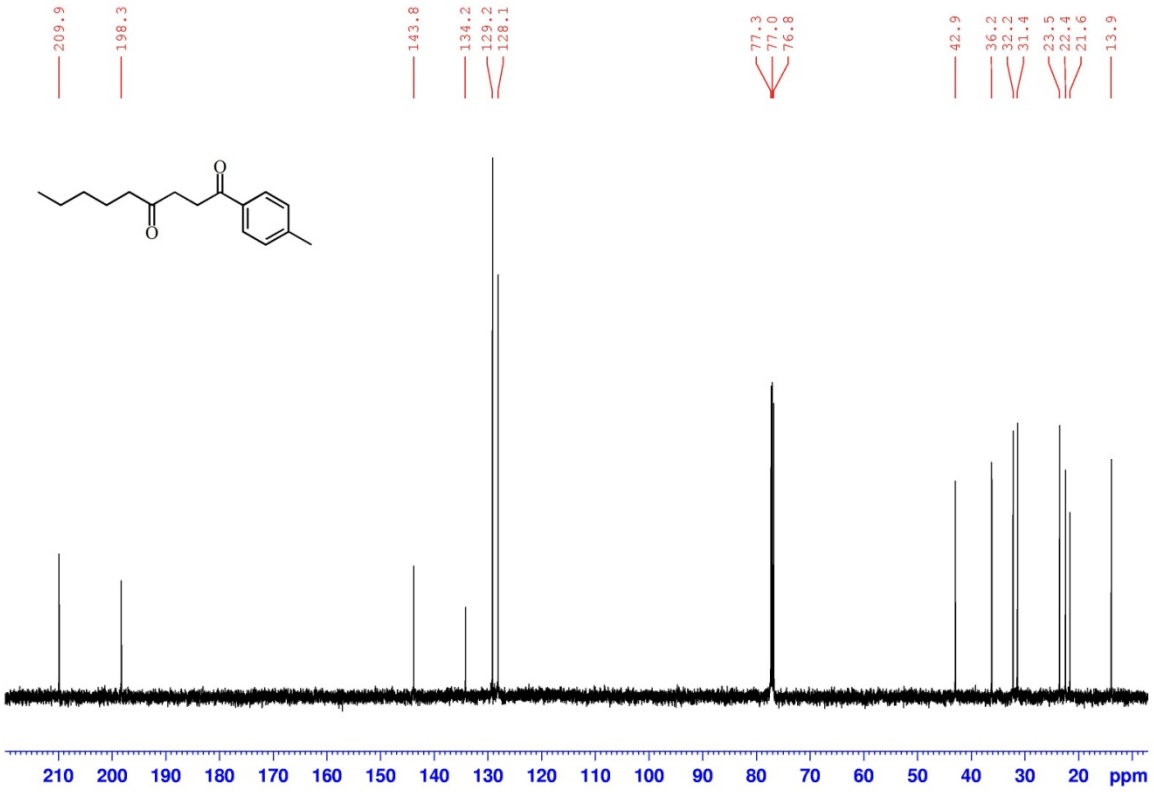
198.2
144.0
141.1
134.1
129.3
128.5
128.3
128.2
126.1
77.3
77.0
76.8
44.5
36.4
32.3
29.8
21.7
Ph
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm


7.89
7.87
7.25
7.24
3.26
3.25
3.24
2.85
2.84
2.83
2.53
2.52
2.50
2.40
1.65
1.63
1.62
1.60
1.59
1.35
1.33
1.32
1.31
1.30
1.30
1.28
1.27
1.27
1.26
0.91
0.89
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
2.10
2.00
1.99
2.00
3.09
2.06
4.13
3.05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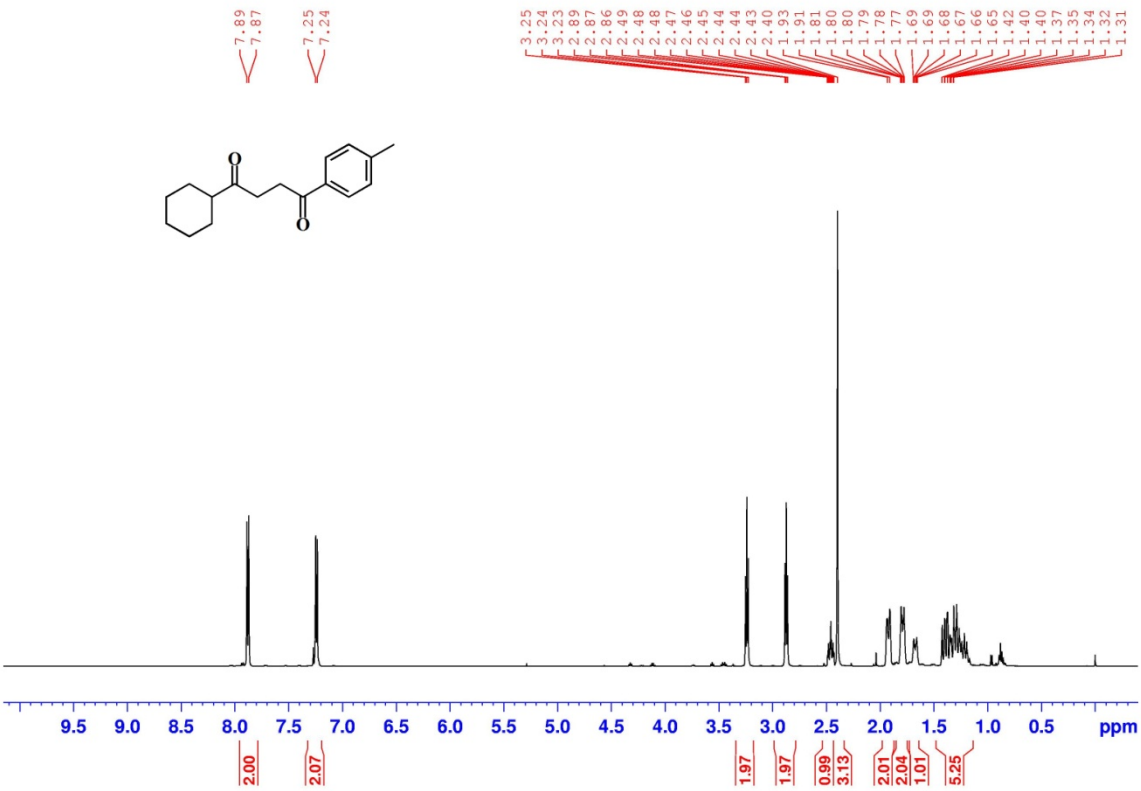
198.3
143.8
134.2
129.2
128.1
77.3
77.0
76.8
42.9
36.2
32.2
31.4
23.5
22.4
21.6
13.9
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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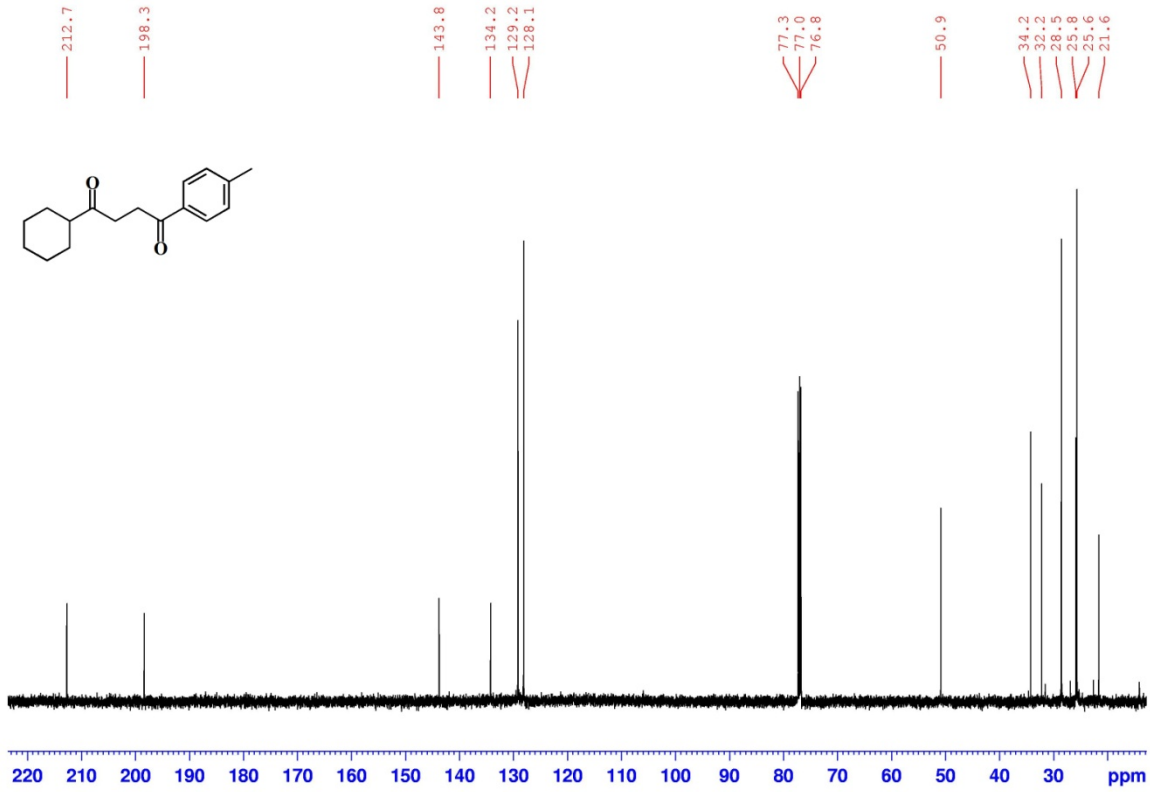
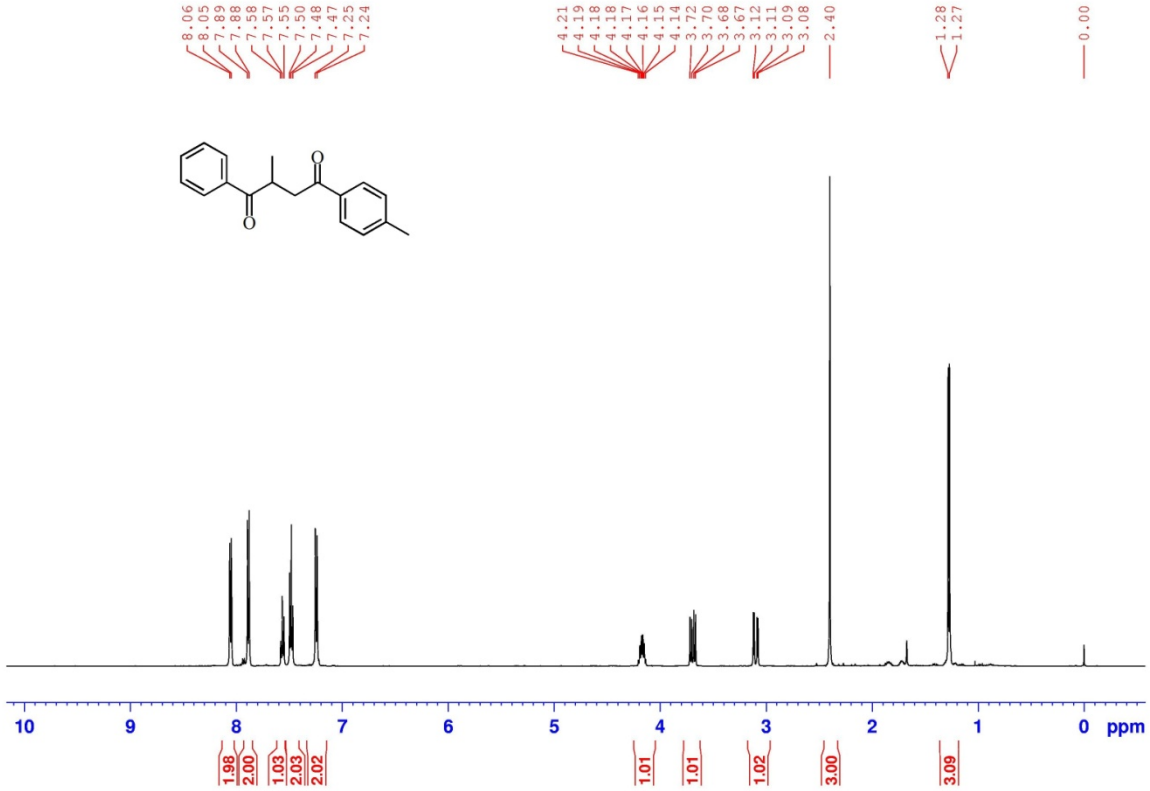
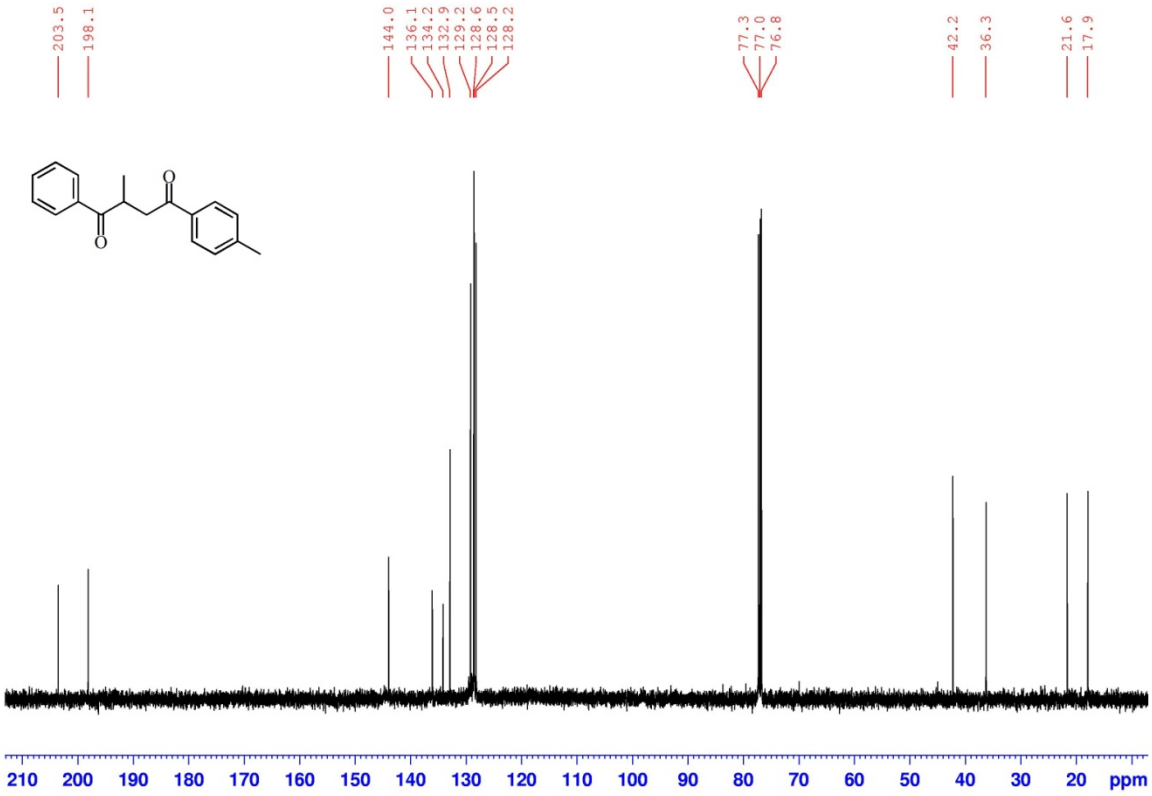
7.87
7.25
7.24
3.25
3.24
3.23
2.89
2.87
2.86
2.49
2.48
2.48
2.47
2.46
2.45
2.44
2.44
2.43
2.40
1.93
1.91
1.81
1.80
1.80
1.79
1.78
1.77
1.69
1.69
1.68
1.67
1.66
1.65
1.42
1.40
1.40
1.37
1.35
1.34
1.32
1.31
O
O
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.00
2.07
1.97
1.97
0.99
3.13
2.01
2.04
1.01
5.25


212.7
198.3
143.8
134.2
129.2
128.1
77.3
77.0
76.8
50.9
34.2
32.2
28.5
25.8
25.6
21.6
O
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm


8.06
8.05
7.89
7.88
7.58
7.57
7.55
7.50
7.48
7.47
7.25
7.24
4.21
4.19
4.18
4.18
4.17
4.16
4.15
4.14
3.72
3.70
3.68
3.67
3.12
3.11
3.09
3.08
2.40
1.28
1.27
0.00
10
9
8
7
6
5
4
3
2
1
0
ppm
1.98
2.00
1.03
2.03
2.02
1.01
1.01
1.02
3.00
3.09


203.5
198.1
144.0
136.1
134.2
132.9
129.2
128.6
128.5
128.2
77.3
77.0
76.8
42.2
36.3
21.6
17.9
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm